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Kavesh et al.

[11] 4,413,110
[45] Nov. 1, 1983

[54] **HIGH TENACITY, HIGH MODULUS POLYETHYLENE AND POLYPROPYLENE FIBERS AND INTERMEDIATES THEREFORE**

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[21] Appl. No.: **359,019**

[22] Filed: **Mar. 19, 1982**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 259,266, Apr. 30, 1981, abandoned.

[51] **Int. Cl.**[3] **C08F 10/02**; C08F 10/06; D01F 6/04; D01F 6/06

[52] **U.S. Cl.** **526/348.1**; 264/164; 264/205; 264/210.8; 524/108; 524/366; 524/462; 524/464; 524/583; 524/585; 526/351; 526/352

[58] **Field of Search** 526/348.1, 351, 352

[56] **References Cited**

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4,137,394 1/1979 Meihuizen et al. 528/502

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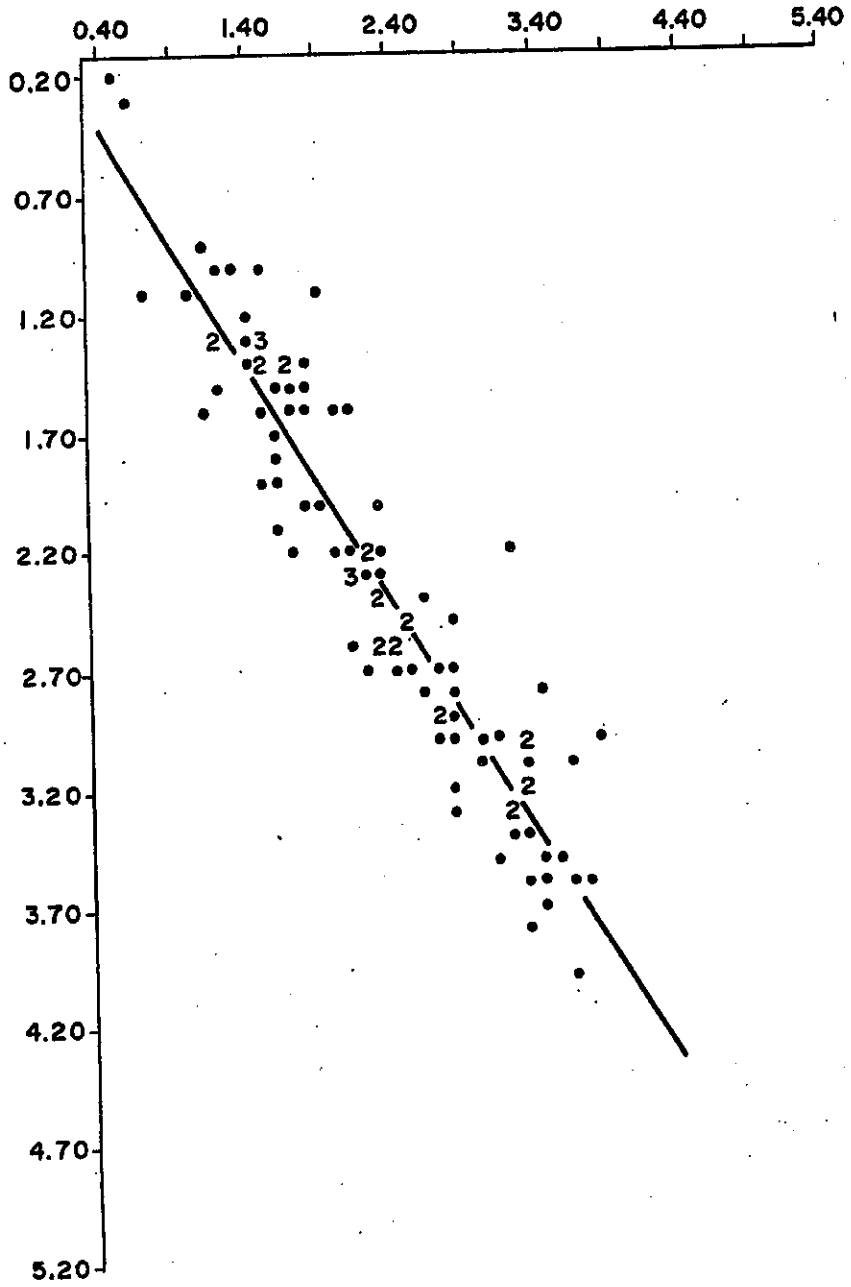
Attorney, Agent, or Firm—Alan M. Doernberg; Gerhard H. Fuchs; Roy H. Massengill

[57] **ABSTRACT**

Solutions of ultrahigh molecular weight polymers such as polyethylene in a relatively non-volatile solvent are extruded through an aperture at constant concentration through the aperture and cooled to form a first gel of indefinite length. The first gels are extracted with a volatile solvent to form a second gel and the second gel is dried to form a low porosity xerogel. The first gel, second gel or xerogel, or a combination, are stretched. Among the products obtainable are polyethylene fibers of greater than 30 or even 40 g/denier tenacity and of modulus greater than 1000 or even 1600 or 2000 g/denier.

19 Claims, 7 Drawing Figures

FIG. 1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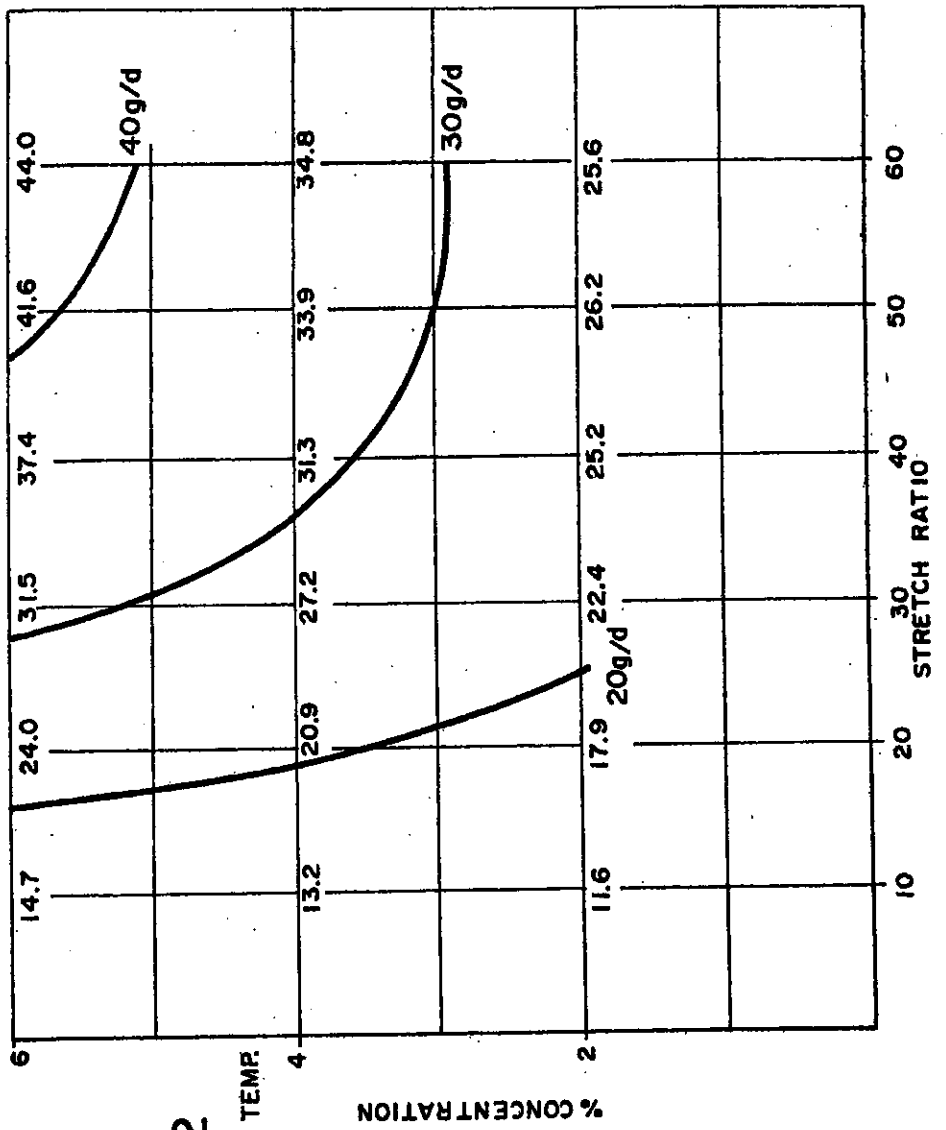

FIG. 2

140°C = STRETCH TEMP.
24 IV PE

FIG. 3

4% GEL CONCENTRATION
24 IV

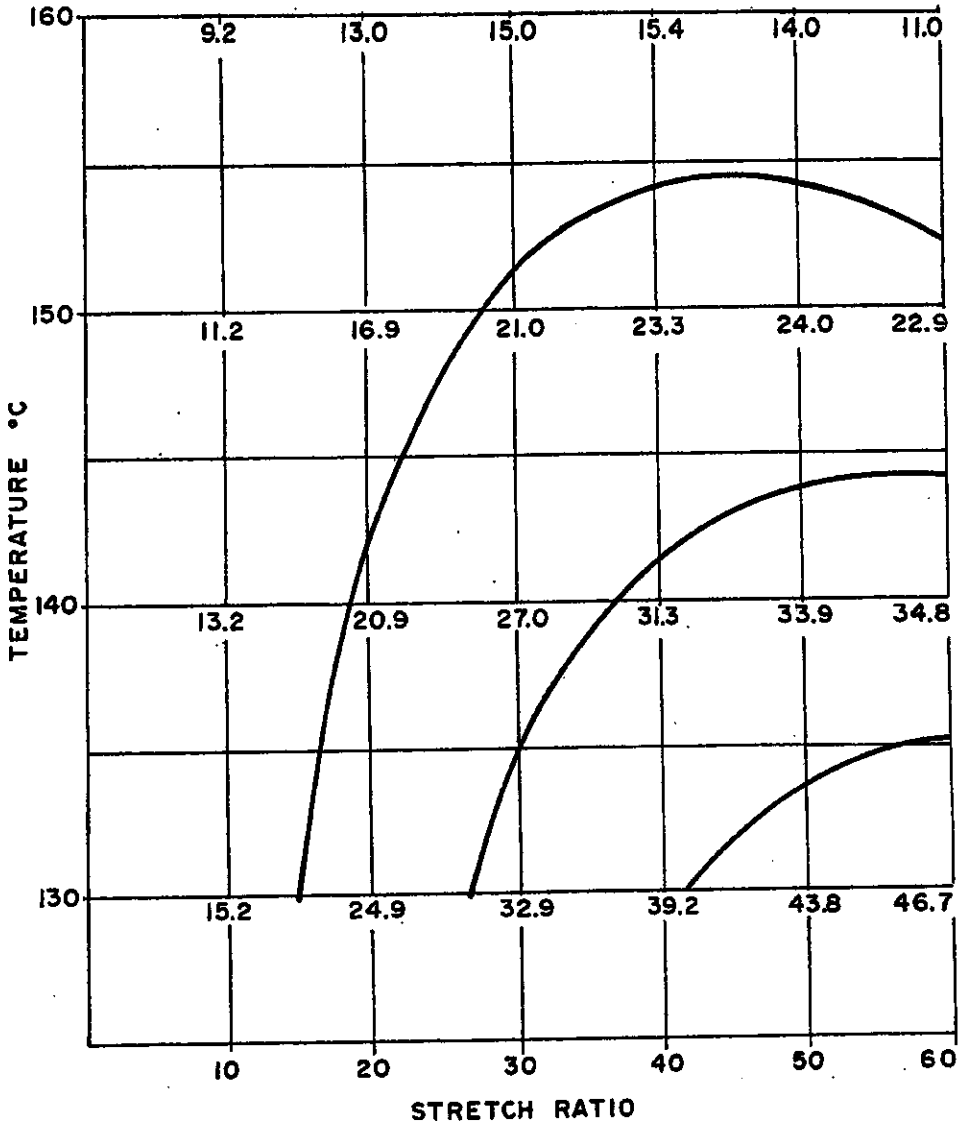

FIG. 4

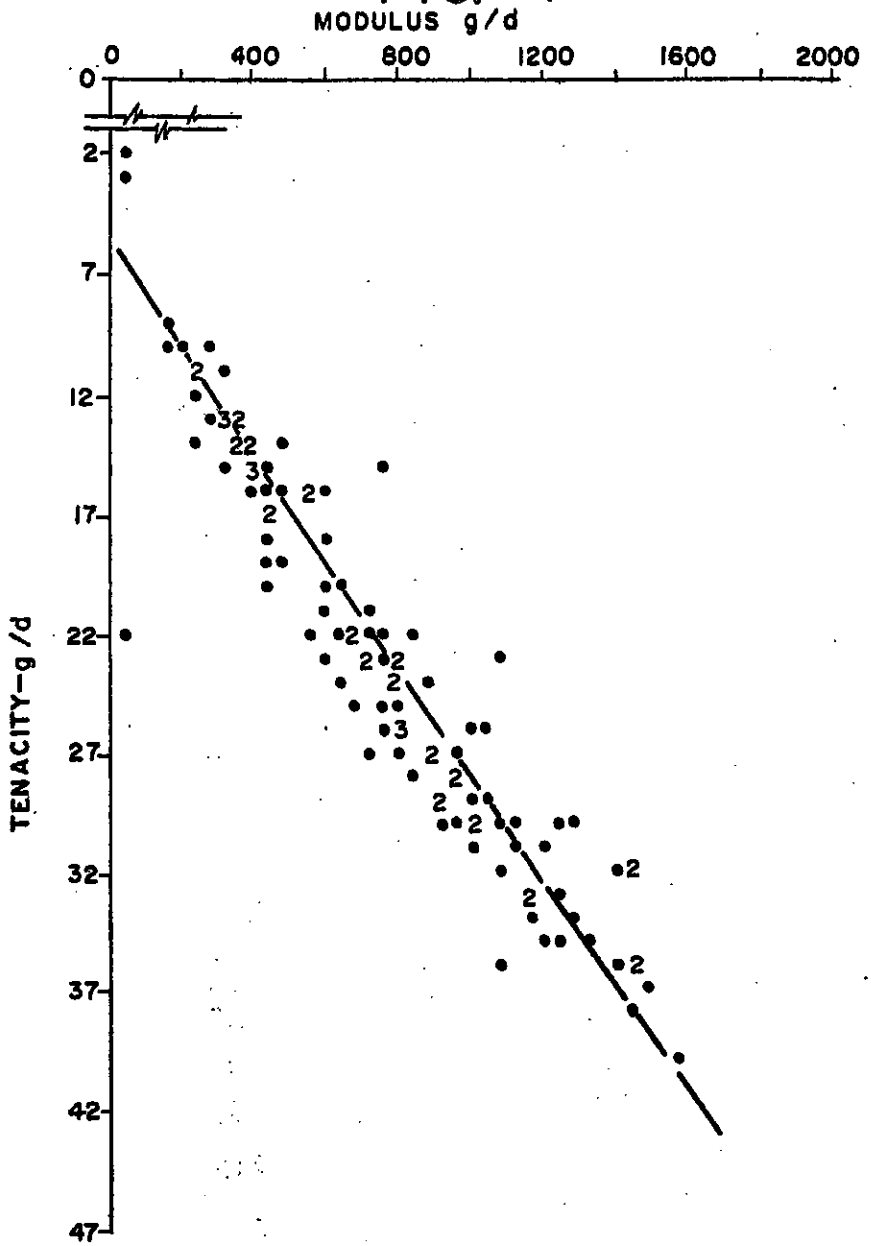
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FIG. 5

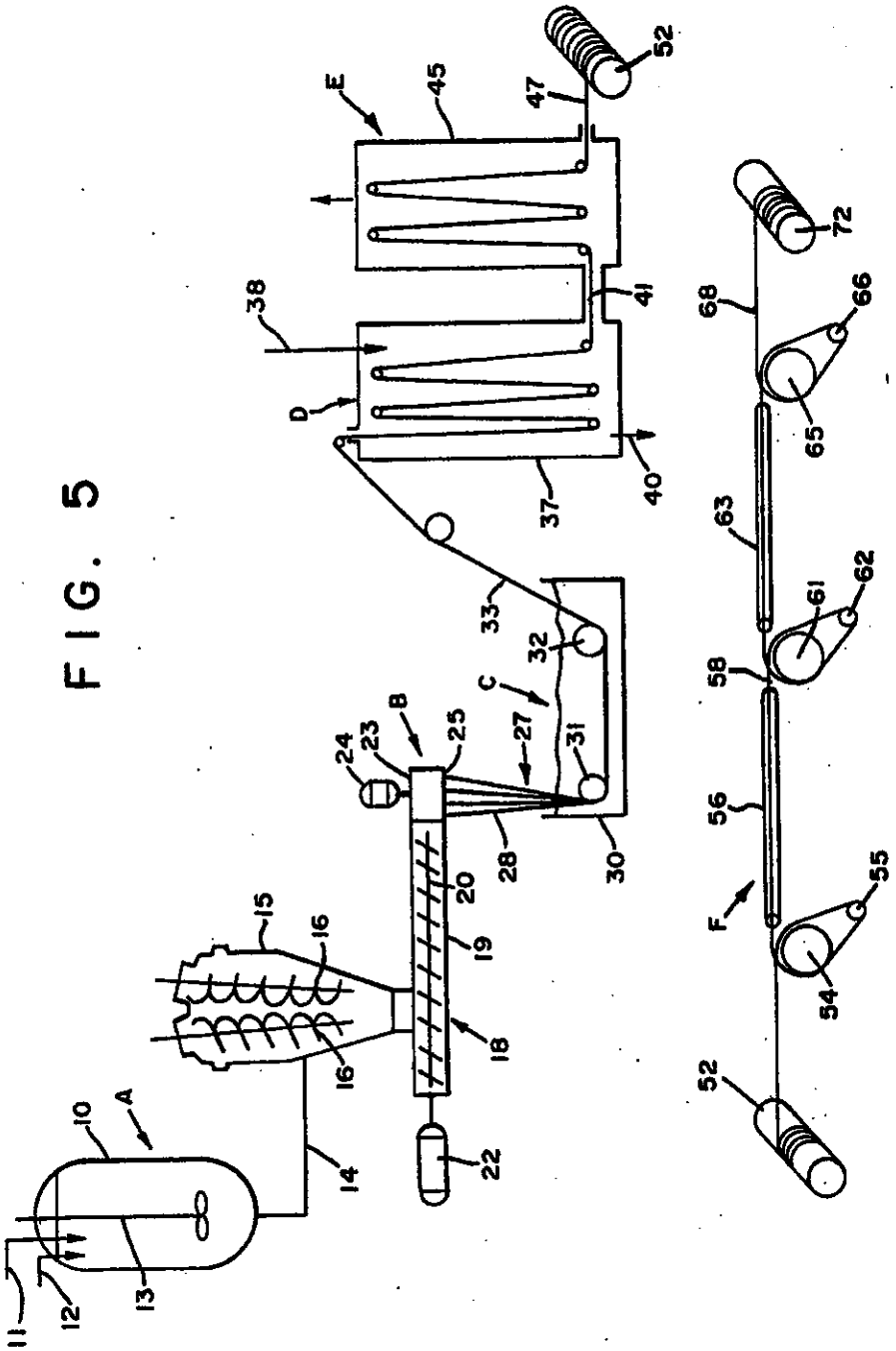
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FIG. 6

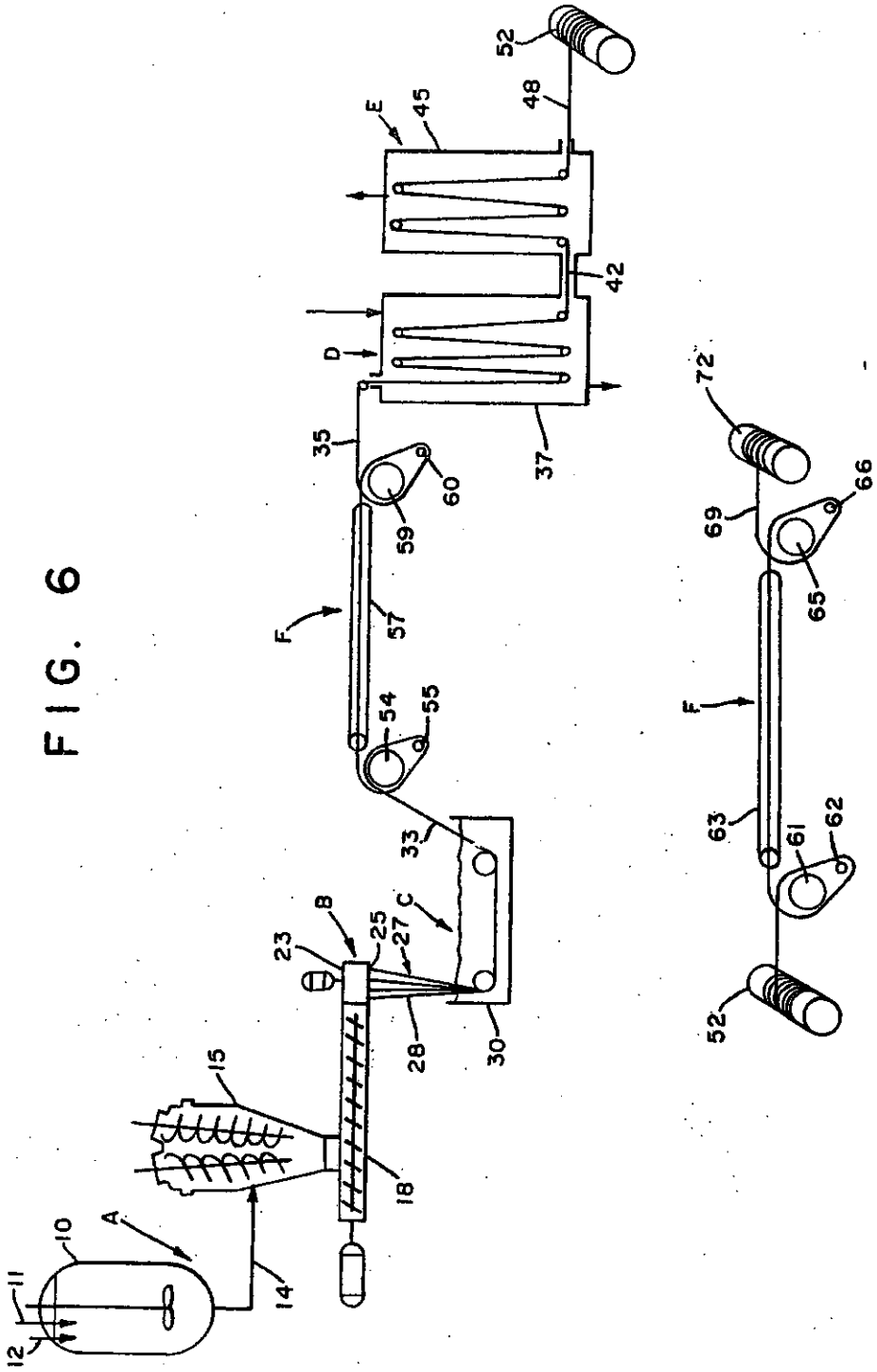
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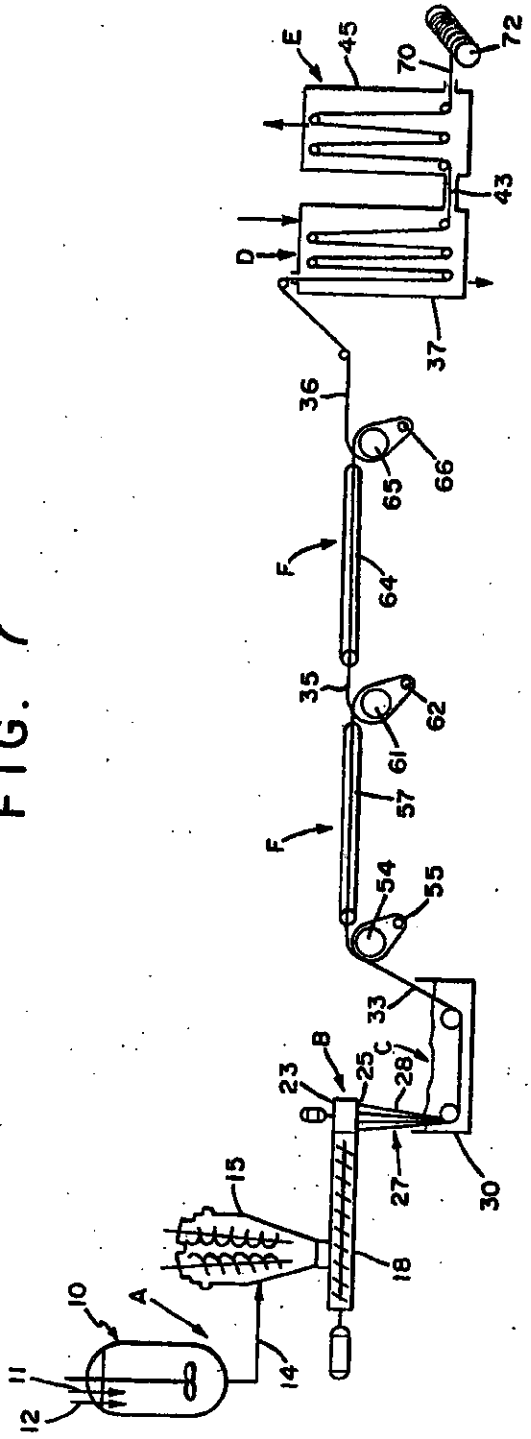
FIG. 7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HIGH TENACITY, HIGH MODULUS POLYETHYLENE AND POLYPROPYLENE FIBERS AND INTERMEDIATES THEREFORE

DESCRIPTION

This is a continuation-in-part of Ser. No. 259,266, filed Apr. 30, 1981, now abandoned.

BACKGROUND OF THE INVENTION

The present invention relates to ultrahigh molecular weight polyethylene and polypropylene fibers having high tenacity, modulus and toughness values and a process for their production which includes a gel intermediate.

The preparation of high strength, high modulus polyethylene fibers by growth from dilute solution has been described by U.S. Pat. No. 4,137,394 to Meihuizen et al. (1979) and pending application Ser. No. 225,288 filed Jan. 15, 1981, now U.S. Pat. No. 4,356,138.

Alternative methods to the preparation of high strength fibers have been described in various recent publications of P. Smith, A. J. Pennings and their coworkers. German Off. No. 3004699 to Smith et al. (Aug. 21, 1980) describes a process in which polyethylene is first dissolves in a volatile solvent, the solution is spun and cooled to form a gel filament, and finally the gel filament is simultaneously stretched and dried to form the desired fiber.

UK Patent application GB No. 2,051,667 to P. Smith and P. J. Lemstra (Jan. 21, 1981) discloses a process in which a solution of the polymer is spun and the filaments are drawn at a stretch ratio which is related to the polymer molecular weight, at a drawing temperature such that at the draw ratio used the modulus of the filaments is at least 20 GPa. The application notes that to obtain the high modulus values required, drawing must be performed below the melting point of the polyethylene. The drawing temperature is in general at most 135° C.

Kalb and Pennings in Polymer Bulletin, vol. 1, pp. 879–80 (1979), J. Mat. Sci., vol. 15, 2584–90 (1980) and Smook et at. in Polymer Bull., vol. 2, pp. 775–83 (1980) describe a process in which the polyethylene is dissolved in a nonvolatile solvent (paraffin oil) and the solution is cooled to room temperature to form a gel. The gel is cut into pieces, fed to an extruder and spun into a gel filament. The gel filament is extracted with hexane to remove the paraffin oil, vacuum dried and the stretched to form the desired fiber.

In the process described by Smook et. al. and Kalb and Pennings, the filaments were non-uniform, were of high porosity and could not be stretched continuously to prepare fibers of indefinite length.

BRIEF DESCRIPTION OF THE INVENTION

The present invention includes a stretched polyethylene fiber of substantially indefinite length being of weight avarage molecular weight at least about 500,000 and having a tenacity of at least about 20 g/denier, a tensile modulus at least about 500 g/denier, a creep value no more than about 5% (when measured at 10% of breaking load for 50 days at 23° C.), a porosity less than about 10% and a melting temperature of at least about 147° C. measured at 10° C./minute heating rate by differential scanning calorimetry).

The present invention also includes a stretched polyethylene fiber of substantially indefinite length being of

weight average molecular weight of at least about 1,000,000 and having a tensile modulus of at least about 1600 g/denier, a main melting point of at least about 147° C. (measured at 10° C./minute heating rate by differential scanning calorimetry) and an elongation-to-break of not more than 5%.

The present invention also includes a stretched polypropylene fiber of substantially indefinite length being of weight average molecular weight of at least about 750,000 and having a tenacity of at least about 8 g/denier, a tensile modulus of at least about 160 g/denier and a main melting temperature of at least about 168° C. (measured at 10° C./minute heating rate by differential scanning calorimetry)

The present invention also includes a polyolefin gel fiber of substantially indefinite length comprising between about 4 and about 20 weight % solid polyethylene of weight average molecular weight at least about 500,000 or solid polypropylene of weight average molecular weight at least about 750,000, and between about 80 and about 96 weight % of a swelling solvent miscible with high boiling hydrocarbon and having an atmospheric boiling point less than about 50° C.

The preferred method of preparing the novel polyethylene and polypropylene fibers of the present invention is via the novel polyolefin gel fiber of the invention and, more preferably, also via a novel xerogel fiber, by a process claimed in out copending, commonly assigned application Ser. No. 539,020, filed herewith.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graphic view of the tenacities of polyethylene fibers prepared according to Examples 3–99 of the present invention versus calculated values therefore as indicated in the Examples. The numbers indicate multiple points.

FIG. 2 is a graphic view of the calculated tenacities of polyethylene fibers prepared according to Examples 3–99 as a function of polymer concentration and draw ratio at a constant temperature of 140° C.

FIG. 3 is a graphic view of the calculated tenacities of polyethylene fibers prepared according to Examples 3–99 as a function of draw temperature and draw (or stretch) ratio at a constant polymer concentration of 4%.

FIG. 4 is a graphic view of tenacity plotted against tensile modulus for polyethylene fibers prepared in accordance with Examples 3–99.

FIG. 5 is a schematic view of a first process used to prepare the products of the present invention.

FIG. 6 is a schematic view of a second process used to prepare the products of the present invention.

FIG. 7 is a schematic view of a third process used to prepare th products of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

There are many applications which require a load bearing element of high strength, modulus, toughness, dimensional and hydrolytic stability and high resistance to creep under sustained loads.

For example, marine ropes and cables, such as the mooring lines used to secure supertankers to loading stations and the cables used to secure deep sea drilling platforms to underwater anchorage, are presently constructed of materials such as nylon, polyester, aramids and steel which are subject to hydrolytic or corrosive

attack by sea water. In consequence such mooring lines and cables are construted with significant safety factors and are replaced frequently. The greatly increased weight and the need for frequent replacement create substantial operational and economic burdens.

The fibers and films of this invention are of high strength, extraordinarily high modulus and great toughness. They are dimensionally and hydrolytically stable and resistant to creep under sustained loads.

The fibers and films of the invention prepared according to the present process possess these properties in a heretofore unattained combination, and are therefore quite novel and useful materials.

Other applications for the fibers and films of this invention include reinforcements in thermoplastics, thermosetting resins, elastomers and concrete for uses such as pressure vessels, hoses, power transmission belts, sports and automotive equipment, and building construction.

In comparison to the prior art fibers perpared by Smith, Lemstra and Pennings described in Off No. 30 04 699, GB No. 205,1667 and other cited references, the strongest fibers of the present invention are of higher melting point, higher tenacity and much higher modulus. Additionally, they are more uniform, and less porous than the prior art fibers.

In comparison with Off No. 30 04 699 to Smith et. al. the process of the present invention has the advantage of greater controllability and reliability in that the steps of drying and stretching may be separate and each step may be carried out under otimal conditions. To illustrate, Smith & Lemstra in Polymer Bulletin, vol. 1, pp. 733–36 (1979) indicate that drawing temperature, below 143° C., had no effect on the relationships between either tenacity or modulus and stretch ratio. As will be seen, the properties of the fibers of the present invention may be controlled in part by varying stretch temperature with other factors held constant.

In comparison with the procedures described by Smook et. al in Polymer Bulletin, vol. 2, pp. 775–83 (1980) and in the above Kalb and Pennings articles, the process of the present invention has the advantage that the intermediate gel fibers which are spun are of uniform concentration and this concentration is the same as the polymer solution as prepared. The advantages of this unformity are illustrated by the fact that the fibers of the present invention may be stretched in a continuous operation to prepare packages of indefinite length. Additionally, the intermediate xerogel fibers of the present invention preferably contain less than about 10 volume % porosity compared to 23–65% porosity in the dry gel fibers described by Smook et. al. and Kalb and Pennings.

The crystallizable polymer used in the present invention may be polyethylene or polypropylene. In the case of polyethylene, suitable polymers have molecular weights (by intrinsic viscosity) in the range of about one to ten million. This corresponds to a weight average chain length of 3.6×10^4 to 3.6×10^5 monomer units or 7×10^4 to 7.1×10^5 carbons. Polypropylene should have similar backbone carbon chain lengths. The weight average molecular weight of polyethylene used is at least about 500,000 (6 IV), preferably at least about 1,000,000 (10 IV) and morre preferably between about 2,000,000 (16 IV) and about 8,000,000 (42 IV). The weight average molecular weight of polypropylene used is at least about 750,000 (5 IV), preferably at least about 1,000,000 (6 IV), more preferably at least about 1,500,000 (9 IV), and most preferably between about 2,000,000 (11 IV) and about 8,000,000 (33 IV). The IV numbers represent intrinsic visosity of the polymer in decalin at 135° C.

The first solvent should be non-volatile under the processing conditions. This is necessary in order to maintain essentially constant the concentration of solvent upstream and through the aperture (die) and to prevent non-uniformity in liquid content of the gel fiber or film containing first solvent. Preferably, the vapor pressure of the first solvent should be no more than about 20 kPa (about one-fifth of an atmosphere) at 175° C., or at the first temperature. Preferred first solvents for hydrocarbon polymers are aliphatic and aromatic hydrocarbons of the desired non-volatility and solubility for the polymer. The polymer may be present in the first solvent at a first concentration which is selected from a relatively narrow range, e.g. about 2 to 15 weight percent, preferably about 4 to 10 weight percent and more preferably about 5 to 8 weight percent; however, once chosen, the concentration should not vary adjacent the die or otherwise prior to cooling to the second temperature. The concentration should also remain reasonably constant over time (i.e. length of the fiber or film).

The first temperature is chosen to achieve complete dissolution of the polymer in the first solvent. The first temperature is the minimum temperature at any point between where the solution is formed and the die face, and must be greater than the gelation temperature for the polymer in the solvent at the first concentration. For polyethylene in paraffin oil at 5-15% concentration, the gelation temperature is approximately 100–130° C.; therefore, a preferred first temperature can be between 180° C. and 250° C., more preferably 200–240° C. While temperatures may vary above the first temperature at various points upstream of the die face, excessive temperatures causative of polymer degradation should be avoided. To assure complete solubility, a first temperature is chosen whereat the solubility of the polymer exceeds the first concentration, and is typically at least 100% greater. The second temperature is chosen whereas the solubility of the polymer is much less than the first concentration. Preferably, the solubility of the polymer in the first solvent at the second temperature is no more than 1% of the first concentration. Cooling of the extruded polymer solution from the first temperature to the second temperature should be accomplished at a rate sufficiently rapid to form a gel fiber which is of substantially the same polymer concentration as existed in the polymer solution. Preferably the rate at which the extruded polymer solution is cooled from the first temperature to the second temperature should be at least about 50° C. per minute.

Some stretching during cooling to the second temperature is not excluded from the present invention, but the total stretching during this stage should not normally exceed about 2:1, and preferably no more than about 1.5:1. As a result of those factors the gel fiber formed upon cooling to the second temperature consists of a continuous polymeric network highly swollen with solvent. The gel usually has regions of high and low polymer density on a microscopic level but is generally free of large (greater than 500 nm) regions void of solid polymer.

An aperture of circular cross section (or other cross section without a major axis in the plane perpendicular to the flow direction more than 8 times the smallest axis

in the same plane, such as oval, Y- or X-shaped aperature) is used so that both gels will be gel fibers, the xerogel will be an xerogel fiber and the product will be a fiber. The diameter of the aperture is not critical, with representative aperatures being between about 0.25 mm and about 5 mm in diameter (or other major axis). The length of the aperture in the flow direction should normally be at least about 10 times the diameter of the aperture (or other similar major axis), perferably at least 15 times and more preferably at least 20 times the diameter (or other similar major axis).

The extraction with second solvent is conducted in a manner that replaces the first solvent in the gel with second solvent without significant changes in gel structure. Some swelling or shrinkage of the gel may occur, but preferably no substantial dissolution, coagulation or precipitation of the polymer occurs.

When the first solvent is a hydrocarbon, suitable second solvents include hydrocarbons, chlorinated hydrocarbons, chlorofluorinated hydrocarbons and others, such as pentane, hexane, heptane, toluene, methylene chloride, carbon tetrachloride, trichlorotrifluoroethane (TCTFE), diethyl ether and dioxane.

The most preferred second solvents are methylene chloride (B.P. 39.8° C.) and TCFE (B.P. 47.5° C.). Preferred second solvents are the non-flammable volatile solvents having an atmospheric boiling point below about 80° C., more preferably below about 70° C. and most preferably below about 50° C. Conditions of extraction should remove the first solvent to less than 1% of the total solvent in the gel.

A preferred combination of conditions is a first temperature between about 150° C. and about 250° C., a second temperature between about −40° C. and about 40° C. and a cooling rate between the first temperature and the second temperature at least about 50° C./minute. It is preferred that the first solvent be a hydrocarbon, when the polymer is a polyolefin such as ultrahigh molecular weight polyethylene. The first solvent should be substantially non-volatile, one measure of which is that its vapor pressure at the first temperature should be less than one-fifth atmosphere (20 kPa), and more preferably less than 2 kPa.

In choosing the fiirst and second solvents, the primary desired difference relates to volatility as discussed above. It is also preferred that the polymers be less soluble in the second solvent at 40° C. than in the first solvent at 150° C.

Once the gel containing second solvent is formed, it is then dried under conditions where the second solvent is removed leaving the solid network of polymer substantially intact. By analogy to silica gels, the resultant material is called herein a "xerogel" meaning a solid matrix corresponding to the solid matrix of a wet gel, with the liquid replaced by gas (e.g. by an inert gas such as nitrogen or by air). The term "xerogel" is not intended to delineate any particular type of surface area, porosity or pore size.

A comparison of the xerogel fibers of the present invention with corresponding dried gel fibers prepared according to prior art indicates the following major differences in structure: The dried xerogel fibers of the present invention preferably contain less than about ten volume percent pores compared to about 55 volume percent pores in the Kalb and Pennings dried gel fibers and about 23–65 volume percent pores in the Smook et al. dried gel fibers. The dried xerogel fibers of the present invention show a surface area (by the B.E.T. technique) of less than about 1 m^2/g as compared to 28.8 m^2/g in a fiber prepared by the prior art method (see Comparative Example 1 and Example 2, below).

The xerogel fibers of the present invention are also novel compared to dry, unstretched fibers of GB No. 2,051,667 and Off. 3004699 and related articles by Smith and Lemstra. This difference is evidenced by the deleterious effect of stretching below 75° C. or above 135° C. upon the Smith and Lemstra unstretched fibers. In comparison, stretching of the present xerogel fibers at room temperature and above 135° C. has beneficial rather than deleterious effects (see, for example, Examples 540–542, below). While the physical nature of these differences are not clear because of lack of information about Smith and Lemstra's unstretched fibers, it appears that one or more of the following characteristics of the present xerogel fibers must be lacking in Smith and Lemstra's unstretched fibers: (1) a crystalline orientation function less than 0.2, and preferably less than 0.1 as measured by wide angle X-ray diffraction; (2) microporosity less than 10% and preferrably less than 3%; (3) a crystallinity index as measured by wide angle X-ray diffraction (see P. H. Hermans and A. Weidinger, Macromol. Chem. vol. 44, p. 24 (1961)) less than 80% and preferably less than 75% (4) no detectable fraction of the triclinic crystalline form and (5) a fractional variation in spherulite size across a diameter of the fiber less than 0.25.

Stretching may be performed upon the gel fiber after cooling to the second temperature or during or after extraction. Alternatively, stretching of the xerogel fiber may be conducted, or a combination of gel stretch and xerogel stretch may be performed. The stretching may be conducted in a single stage or it may be conducted in two or more stages. The first stage stretching may be conducted at room temperatures or at an elevated temperature. Preferably the stretching is conducted in two or more stages with the last of the stages performed at a temperature between about 120° C. and 160° C. Most preferably the stretching is conducted in at least two stages with the last of the stages performed at a temperature between about 135° C. and 150° C. The Examples, and especially Examples 3–99 and 111–486, illustrate how the stretch ratios can be related to obtaining particular fiber properties.

The product polyethylene fibers produced by the present process represent novel articles in that they include fibers with a unique combination of properties: a tensile modulus at least about 500 g/denier (preferably at least about 1000 g/denier, more preferably at least about 1600 g/denier and most preferably at least about 2000 g/denier), a tenacity at least about 20 g/denier (preferably at least about 30 g/denier and more preferably at least about 40 g/denier), a main melting temperature (measured at 10° C./minute heating rate by differential scanning calorimetry) of at least about 147° C. (preferably at least about 149° C.), a porosity of no more than about 10% (preferably no more than about 6% and more preferably no more than about 3%) and a creep value no more than about 5% (preferably no more than about 3%) when measured at 10% of breaking load for 50 days at 23° C. Preferably the fiber has an elongation to break at most about 7,% and more preferably not more than about 5% (which correlates with the preferred tensile modulus of at least about 1000 g/denier). In addition, the fibers have high toughness and uniformity. Furthermore, as indicated in Examples 3–99 and 111–489 below, trade-offs between various properties

can be made in a controlled fashion with the present process.

The novel polypropylene fibers of the present invention also include a unique combination of properties, previously unachieved for polypropylene fibers: a tenacity of at least about 8 g/denier (preferably at least about 11 g/denier and more preferably at least about 13 g/denier), a tensile modulus at least about 160 g/denier (preferably at least about 200 g/denier and more preferably at least about 220 g/denier), a main melting temperature (measured at 10° C./minute heating rate by differential scanning calorimetry) at least about 168° C. (preferably at least about 170° C.) and a porosity less about 10% (preferably no more than about 5%). Preferably, the polypropylene fibers also have an elongation to break less than about 20%.

Additionally a novel class of fibers of the invention are polypropylene fibers possessing a modulus of at least about 220 g/denier, preferably at least about 250 g/denier.

The gel fibers containing first solvent, gel fibers containing second solvent and xerogel fibers of the present invention also represent novel articles of manufacture, distinguished from somewhat similar products described by Smook et al. and by Kalb and Pennings in having a volume porosities of 10% or less compared to values of 23%–65% in the references.

In particular the second gel fibers differ from the comparable prior art materials in having a solvent with an atmospheric boiling point less than about 50° C. As indicated by Examples 100–108, below, the uniformity and cylindrical shape of the xerogel fibers improved progressively as the boiling point of the second solvent declined. As also indicated in Examples 100–108 (see Table III), substantially higher tenacity fibers were produced under equivalent drying and stretching conditions by using trichlorotrifluoroethane (boiling point 47.5° C.) as the second solvent compared to fibers produced by using hexane (boiling point 68.7° C.) as second solvent. The improvement in final fiber is then directly attributable to changes in the second solvent in the second gel fiber. Preferred such second solvents are halogenated hydrocarbons of the proper boiling point such as methylene chloride (dichloromethane) and trichlorotrifluoroethane, with the latter being most preferred.

DESCRIPTION OF THE PREFERRED EMBODIMENT

FIG. 5 illustrates in schematic form a first process to produce the novel fibers, wherein the stretching step F is conducted in two stages on the novel xerogel fiber subsequent to drying step E. In FIG. 5, a first mixing vessel **10** is shown, which is fed with an ultra high molecular weight polymer **11** such as polyethylene of weight average molecular weight at least 500,000 and preferably at least 1,000,000, and to which is also fed a first, relatively non-volatile solvent **12** such as paraffin oil. First mixing vessel **10** is equipped with an agitator **13**. The residence time of polymer and first solvent in first mixing vessel **10** is sufficient to form a slurry containing some dissolved polymer and some relatively finely divided polymer particles, which slurry is removed in line **14** to an intensive mixing vessel **15**. Intensive mixing vessel **15** is equipped with helical agitator blades **16**. The residence time and agitator speed in intensive mixing vessel **15** is sufficient to convert the slurry into a solution. It will be appreciated that the temperature in intensive mixing vessel **15**, either because of external heating, heating of the slurry **14**, heat generated by the intensive mixing, or a combination of the above is sufficiently high (e.g. 200° C.) to permit the polymer to be completely dissolved in the solvent at the desired concentration (generally between about 6 and about 10 percent polymer, by weight of solution). From the intensive mixing vessel **15**, the solution is fed to an extrusion device **18**, containing a barrel **19** within which is a screw **20** operated by motor **22** to deliver polymer solution at reasonably high pressure to a gear pump and housing **23** at a controlled flow rate. A motor **24** is provided to drive gear pump **23** and extrude the polymer solution, still hot, through a spinnerette **25** comprising a plurality of apertures, which may be circular, X-shaped, or, oval-shaped, or in any of a variety of shapes having a relatively small major axis in the plane of the spinnerette when it is desired to form fibers, and having a rectangular or other shape with an extended major axis in the plane of the spinnerette when it is desired to form films. The temperature of the solution in the mixing vessel **15**, in the extrusion device **18** and a t the spinnerette **25** should all equal or exceed a first temperature (e.g. 200° C.) chosen to exceed the gellation temperature (approximately 100–130 C. for polyethylene in paraffin oil). The temperature may vary (e.g. 220° C., 210° C. and 200° C.) or may be constant (e.g. 220° C.) from the mixing vessel **15** to extrusion device **18** to the spinnerette **25**. At all points, however, the concentration of polymer in the solution should be substantially the same. The number of apertures, and thus the number of fibers formed, is not critical, with convenient number of apertures being **16**, **120**, or **240**.

From the spinnerette **25**, the polymer solution passes through an air gap **27**, optionally enclosed and filled with an inert gas such as nitrogen, and optionally provided with a flow of gas to facilitate cooling. A plurality of gel fibers **28** containing first solvent pass through the air gap **27** and into a quench bath **30**, so as to cool the fibers, both in the air gap **27** and in the quench bath **30**, to a second temperature at which the solubility of the polymer in the first solvent is relatively low, such that most of the polymer precipitates as a gel material. While some stretching in the air gap **27** is permissible, it is preferably less than about 2:1, and is more preferably much lower. Substantial stretching of the hot gel fibers in air gap **27** is believed highly detrimental to the properties of the ultimate fibers.

It is preferred that the quench liquid in quench bath **30** be water. While the second solvent may be used as the quench fluid (and quench bath **30** may even be integral with solvent extraction device **37** described below), it has been found in limited testing that such a modification impairs fiber properties.

Rollers **31** and **32** in the quench bath **30** operate to feed the fiber through the quench bath, and preferably operate with little or no stretch. In the event that some stretching does occur across rollers **31** and **32**, some first solvent exudes out of the fibers and can be collected as a to layer in quench bath **30**.

From the quench bath **30**, the cool first gel fibers **33** pass to a solvent extraction device **37** where a second solvent, being of relatively low boiling such as trichlorotrfluoroethane, is fed in through line **38**. The solvent outflow in line **40** contains second solvent and essentially all of tthe first solvent brought it with the cool gel fibers **33**, either dissolved or dispersed in the second solvent. Thus the second gel fibers **41** conducted

out of the solvent extraction device 37 contain substantially only second solvent, and relatively little first solvent. The second gel fibers 41 may have shrunken somewhat compared to the first gel fibers 33, but otherwise contain substantially the same polymer morphology.

In a drying device 45, the second solvent is evaporated from the second gel fibers 41 forming essentially unstretched xerogel fibers 47 which are taken up on spool 52.

From spool 52, or from a plurality of such spools if it is desired to operate the stretching line at a slower feed rate than the take up of spool 52 permits, the fibers are fed over driven fed roll 54 and idler roll 55 into a first heated tube 56, which may be rectangular, cylindrical or other convenient shape. Sufficient heat is applied to the tube 56 to cause the internal temperature to be between about 120 and 140° C. The fibers are stretched at a relatively high draw ratio (e.g. 10:1) so as to form partially stretched fibers 58 taken up by driven roll 61 and idler roll 62. From rolls 61 and 62, the fibers are taken through a second heated tube 63, heated so as to be at somewhat higher temperature, e.g. 130–160° C. and are then taken up by driven take-up roll 65 and idler roll 66, operating at a speed suficient to impart a stretch ratio in heated tube 63 as desired, e.g. about 2.5:1. The twice stretched fibers 68 produced in this first embodiment are taken up on take-up spool 72.

With reference to the six process steps of the process, it can be seen that the solution forming step A is conducted in mixers 13 and 15. The extruding step B is conducted with device 18 and 23, and especially through spinnerette 25. The cooling step C is conducted in airgap 27 and quench bath 30. Extraction step D is conducted in solvent extraction device 37. The drying step E is conducted in drying device 45. The stretching step F is conducted in elements 52–72, and especially in heated tubes 56 and 63. It will be appreciated, howrever, that various other parts of the system may also perform some stretching, even at temperatures substantially below thase of heated tubes 56 and 63. Thus, for example, some stretching (e.g. 2:1) may occur within quench bath 30, within solvent extraction device 37, within drying device 45 or between solvent extraction device 37 and drying device 45.

A second process to produce the novel fiber products is illustrated in schematic form by FIG. 6. The solution forming and extruding steps A and B of the second embodiment are substantially the same as those in the first embodiment illustrated in FIG. 5. Thus, polymer and first solvent are mixed in first mixing vessel 10 and conducted as a slurry in line 14 to intensive mixing device 15 operative to form a hot solution of polymer in first solvent. Extrusion device 18 impells the solution under pressure through the gear pump and housing 23 and then through a plurality of apperatures in spinnerette 27. The hot first gel fibers 28 pass through air gap 27 and quench bath 30 so as to form cool first gel fibers 33.

The cool first gel fibers 33 are conducted over driven roll 54 and idler roll 55 through a heated tube 57 which, in general, is longer than the first heated tube 56 illustrated in FIG. 5. The length of heated tube 57 compensates, in general, for the higher velocity of fibers 33 in the second embodiment of FIG. 6 compared to the velocity of xerogel fibers (47) between take-up spool 52 and heated tube 56 in the first embodiment of FIG. 5. The fibers 33 are drawn through heated tube 57 by driven take-up roll 59 and idler roll 60, so as to cause a relatively high stretch ratio (e.g. 10:1). The once-stretched first gel fibers 35 are conducted into extraction device 37.

In the extraction device 37, the first solvent is extracted out of the gel fibers by second solvent and the novel gel fibers 42 containing second solvent are conducted to a drying device 45. There the second solvent is evaporated from the gel fibers; and novel xerogel fibers 48, being once-stretched, are taken up on spool 52.

Fibers on spool 52 are then taken up by driven feed roll 61 and idler 62 and passed through a heated tube 63, operating at the relatively high temperature of between about 130° and 160° C. The fibers are taken up by driven take up roll 65 and idler roll 66 operating at a speed sufficient to impart a stretch in heated tube 63 as desired, e.g. about 2.5:1. The twice-stretched fibers 69 produced in the second embodiment are then taken up on spool 72.

It will be appreciated that, by comparing the embodiment of FIG. 6 with the embodiment of FIG. 5, the stretching step F has been divided into two parts, with the first part conducted in heated tube 57 performed on the first gel fibers 33 prior to extraction (D) and drying (E), and the second part conducted in heated tube 63, being conducted on xerogel fibers 48 subsequent to drying (E).

A third process to produce novel fiber products is illustrated in FIG. 7, with the solution forming step A, extrusion step B, and cooling step C being substantially identical to the first embodiment of FIG. 5 and the second embodiment of FIG. 6. Thus, polymer and first solvent are mixed in first mixing vessel 10 and conducted as a slurry in line 14 to intensive mixing device 15 operative to form a hot solution of polymer in first solvent. Extrusion device 18 impells the solution under pressure through the gear pump and housing 23 and then through a plurality of apperatures in spinnerette 27. The hot first gel fibers 28 pass through air gap 27 and quench bath 30 so as to form cool first gel fibers 33.

The cool first gel fibers 33 are conducted over driven roll 54 and idler roll 55 through a heated tube 57 which, in general, is longer than the first heated tube 56 illustrated in FIG. 5. The length of heated tube 57 compensates, in general, for the higher velocity of fibers 33 in the third embodiment of FIG. 7 compared to the velocity of xerogel fibers (47) between takeup spool 52 and heated tube 56 in the first embodiment of FIG. 5. The first gel fibers 33 are now taken up by driven roll 61 and idler roll 62, operative to cause the stretch ratio in heated tube 57 to be as desired, e.g. 10:1.

From rolls 61 and 62, the once-drawn first gel fibers 35 are conducted into modified heated tube 64 and drawn by driven take up roll 65 and idler roll 66. Driven roll 65 is operated sufficiently fast to draw the fibers in heated tube 64 at the desired stretch ratio, e.g. 2.5:1. Because of the relatively high line speed in heated tube 64, required generally to match the speed of once-drawn gel fibers 35 coming off of rolls 61 and 62, heated tube 64 in the third embodiment of FIG. 7 will, in general, be longer than heated tube 63 in either the second embodiment of FIG. 6 or the first embodiment of FIG. 5. While first solvent may exude from the fiber during stretching in heated tubes 57 and 64 (and be collected at the exit of each tube), the first solvent is sufficiently non-volatile so as not to evaporate to an appreciable extent in either of these heated tubes.

The twice-stretched first gel fiber 36 is then conducted through solvent extraction device 37, where the second, volatile solvent extracts the first solvent out of the fibers. The second gel fibers, containing substantially only second solvent, is then dried in drying device 45, and the twice-stretched fibers 70 are then taken up on spool 72.

It will be appreciated that, by comparing the third embodiment of FIG. 7 to the first two embodiments of FIGS. 5 and 6, the stretching step (F) is performed in the third embodiment in two stages, both subsequent to cooling step C and prior to solvent extracting step D.

The invention will be further illustrated by the examples below. The first example illustrates the prior art techniques of Smook et. al. and the Kalb and Pennings articles.

COMPARATIVE EXAMPLE 1

A glass vessel equipped with a PTFE paddle stirrer was charged with 5.0 wt% linear polyethylene (sold as Hercules UHMW 1900, having 24 IV and approximately 4×10^6 M.W.), 94.5 wt% paraffin oil (J. T. Baker, 345–355 Saybolt viscosity) and 0.5 wt% antioxidant (sold under the trademark Ionol).

The vessel was sealed under nitrogen pressure and heated with stirring to 150° C. The vessel and its contents were maintained under slow agitation for 48 hours. At the end of this period the solution was cooled to room temperature. The cooled solution separated into two phases-A "mushy" liquid phase consisting of 0.43 wt% polyethylene and a rubbery gel phase consisting of 8.7 wt% polyethylene. The gel phase was collected, cut into pieces and fed into a 2.5 cm (one inch) Sterling extruder equipped with a 21/1 L/D polyethylene-type screw. The extruder was operated at 10 RPM, 170° C. and was equipped with a conical single hole spinning die of 1 cm inlet diameter, 1 mm exit diameter and 6 cm length.

The deformation and compression of the gel by the extruder screw caused exudation of paraffin oil from the gel. This liquid backed up in the extruder barrel and was mostly discharged from the hopper end of the extruder. At the exit end of the extruder a gel fiber of approximately 0.7 mm diameter was collected at the rate of 1.6 m/min. The gel fiber consisted of 24–38 wt% polyethylene. The solids content of the gel fiber varied substantially with time.

The paraffin oil was extracted from the extruded gel fiber using hexane and the fiber was dried under vacuum at 50° C. The dried gel fiber had a density of 0.326 g/cm^3. Therefore, based on a density of 0.960 for the polyethylene constituent, the gel fiber consisted of 73.2 volume percent voids. Measurement of pore volume using a mercury porosimeter showed a pore volume of 2.58 cm^3/g. A B.E.T. measurement of surface area gave a value of 28.8 m^2/g.

The dried fiber was stretched in a nitrogen atmosphere within a hot tube of 1.5 meters length. Fiber feed speed was 2 cm/min. Tube temperature was 100° C. at the inlet increasing to 150° C. at the outlet.

It was found that, because of filament nonuniformity, stretch ratios exceeding 30/1 were not sustainable for periods exceeding about 20 minutes without filament breakage.

The properties of the fiber prepared at 30/1 stretch ratio were as follows:

denier—99
tenacity—23 g/d
modulus—980 g/d
elongation at break—3%
work-to-break—6570 in lbs./in^3 (45 MJ/m^3)

The following example is illustrative of the present invention:

EXAMPLE 2

An oil jacketed double helical (Helicone®) mixer constructed by Atlantic Research Corporation was charged with 5.0 wt% linear polyethylene (Hercules UHMW 1900 having a 17 IV and approximately 2.5×10^6 M.W.) and 94.5 wt% paraffin oil (J. T. Baker, 345–355 Saybolt viscosity). The charge was heated with agitation at 20 rpm to 200° C. under nitrogen pressure over a period of two hours. After reaching 200° C., agitation was maintained for an additional two hours.

The bottom discharge opening of the Helicone mixer was fitted with a single hole capillary spinning die of 2 mm diameter and 9.5 mm length. The temperature of the spinning die was maintained at 200° C.

Nitrogen pressure applied to the mixer and rotation of the blades of the mixer were used to extrude the charge through the spinning die. The extruded uniform solution filament was quenched to a gel state by passage through a water bath located at a distance of 33 cm (13 inches) below the spinning die. The gel filament was wound up continuously on a 15.2 cm (6 inch) diameter bobbin at the rate of 4.5 meters/min.

The bobbins of gel fiber were immersed in trichlorotrifloroethane (fluorocarbon 113 or "TCTFE") to exchange this solvent for paraffin oil as the liquid constituent of the gel. The gel fiber was unwound from a bobbin, and the fluorocarbon solvent evaporated at 22°–50° C.

The dried fiber was of 970± 100 denier. The density of the fiber was determined to be 950 kg/m^3 by the density gradient method. Therefore, based on a density of 960 kg/m^3 for the polyethylene constituent, the dried fiber contained one volume percent voids. A B.E.T. measurement of the surface area gave a value less than 1 m^2/g.

The dried gel fiber was fed at 2 cm/min into a hot tube blanketed with nitrogen and maintained at 100° C. at its inlet and 140° C. at its outlet. The fiber was stretched continously 45/1 within the hot tube for a period of three hours without experiencing fiber breakage. The properties of the stretched fiber were:

denier—22.5
tenacity—37.6 g/d
modulus—1460 g/d
elongation—4.1%
work-to-break—12,900 in-lbs/in^3 (89 MJ/m^3)

EXAMPLES 3–99

A series of fiber samples was prepared following the procedures described in Example 2, but with variations introduced in the following material and process parameters:

a. polyethylene IV (molecular weight)
b. polymer gel concentration
c. stretch temperature
d. fiber denier
e. stretch ratio

The results of these experiments upon the final fiber properties obtained are presented in Table I. The Polymer intrinsic viscosity values were 24 in Examples 3–49 and 17 in Examples 50–99. The gel concentration was

2% in Examples 26–41, 4% in Examples 3–17, 5% in Examples 42–99 and 6% in Examples 18–25.

TABLE I

Ex.	Stretch Temp., °C.	Stretch Ratio	Denier	Tenacity g/d	Modulus g/d	Elong %
3	142	15.6	2.8	17.8	455.	6.7
4	145	15.5	2.8	18.6	480.	6.7
5	145	19.6	2.2	19.8	610.	5.2
6	145	13.0	3.4	13.7	350.	6.2
7	145	16.6	2.7	15.2	430.	5.7
8	144	23.9	1.8	23.2	730.	4.9
9	150	16.0	2.7	14.6	420.	5.0
10	150	27.3	1.6	21.6	840.	4.0
11	149	23.8	1.8	21.8	680.	4.6
12	150	27.8	1.6	22.6	730.	4.3
13	140	14.2	3.1	16.5	440.	5.3
14	140	22.0	2.0	21.7	640.	4.7
15	140	25.7	1.7	26.1	810.	4.7
16	140	3.4	5.6	11.2	224.	18.0
17	140	14.9	2.9	20.8	600.	5.6
18	145	19.5	11.7	16.4	480.	6.3
19	145	11.7	19.4	16.3	430.	6.1
20	145	22.3	10.2	24.1	660.	5.7
21	145	47.4	4.8	35.2	1230.	4.3
22	150	15.1	15.0	14.0	397.	6.5
23	150	56.4	4.0	28.2	830.	4.4
24	150	52.8	4.3	36.3	1090	4.5
25	150	12.8	17.8	19.1	440.	7.2
26	143	10.3	21.4	8.7	178.	7.0
27	146	1.8	120.0	2.1	22.	59.7
28	146	3.2	69.5	2.7	37.	40.5
29	145	28.0	7.9	16.0	542.	4.9
30	145	50.2	4.4	21.6	725.	4.0
31	145	30.7	7.2	22.7	812.	4.2
32	145	10.2	21.8	16.2	577.	5.6
33	145	22.3	9.9	15.3	763.	2.8
34	150	28.7	7.7	10.5	230.	8.4
35	150	12.1	18.3	12.6	332.	5.2
36	150	8.7	25.5	10.9	308.	5.9
37	150	17.4	12.7	14.1	471.	4.6
38	140	12.0	18.5	12.7	357.	7.3
39	140	21.5	10.3	16.1	619.	4.2
40	140	36.8	6.0	23.8	875.	4.1
41	140	59.7	3.7	26.2	1031.	3.6
42	145	13.4	25.0	12.9	344.	8.3
43	145	24.4	13.7	22.3	669.	5.9
44	145	25.2	13.3	23.2	792.	4.9
45	145	33.5	10.0	29.5	1005.	4.9
46	150	17.2	19.5	14.2	396.	5.6
47	150	16.0	21.0	15.7	417.	7.2
48	140	11.2	30.0	13.1	316.	8.3
49	140	21.0	16.0	23.0	608.	6.0
50	130	15.8	64.9	14.2	366.	6.0
51	130	44.5	23.1	30.8	1122.	4.4
52	130	24.3	42.4	26.8	880.	4.7
53	130	26.5	38.8	23.6	811.	4.2
54	140	11.0	93.3	14.5	303.	8.4
55	140	28.3	36.3	24.7	695.	4.8
56	140	43.4	23.7	30.3	905.	4.8
57	140	18.4	55.9	19.7	422.	6.6
58	150	15.7	65.5	12.8	337.	8.6
59	150	43.4	23.7	30.9	1210.	4.5
60	150	33.6	30.6	28.9	913.	4.8
61	150	54.4	18.9	30.2	1134.	3.7
62	150	13.6	71.1	10.4	272.	12.2
63	150	62.9	15.4	30.5	1008.	4.0
64	150	26.6	36.4	20.4	638.	7.0
65	150	36.1	26.8	32.0	1081.	5.3
66	150	52.0	18.6	34.0	1172.	4.1
67	150	73.3	13.2	35.3	1314.	3.8
68	140	14.6	66.1	13.9	257.	14.9
69	140	30.1	32.1	28.5	933.	4.5
70	140	45.6	21.2	35.9	1440.	3.9
71	140	43.0	22.5	37.6	1460.	4.1
72	140	32.3	30.1	33.1	1170.	4.3
73	140	57.3	16.9	39.6	1547.	3.8
74	130	16.3	59.4	21.6	556.	5.5
75	130	20.6	47.0	25.6	752.	5.3
76	130	36.3	26.7	33.0	1144.	4.1
77	130	49.4	19.6	30.4	1284.	3.8
78	130	24.5	44.6	26.4	990.	4.5
79	130	28.6	38.2	27.1	975.	4.5
80	130	42.2	25.9	34.7	1200.	4.4
81	140	40.3	27.1	33.2	1260.	4.0
82	140	58.7	18.6	35.5	1400.	4.0
83	145	47.9	22.8	32.1	1460.	4.0
84	145	52.3	20.9	37.0	1500.	4.0
85	130	13.6	80.4	12.8	275.	8.0
86	130	30.0	36.4	24.8	768.	5.0
87	130	29.7	36.8	28.6	1005.	4.5
88	140	52.0	21.0	36.0	1436.	3.5
89	140	11.8	92.3	10.1	151.	18.5
90	140	35.3	31.0	29.8	1004.	4.5
91	140	23.4	46.8	26.6	730.	5.5
92	150	14.6	74.9	11.5	236.	11.0
93	150	35.7	30.6	27.4	876.	4.5
94	150	31.4	34.8	27.0	815.	5.0
95	150	37.8	28.9	29.8	950.	4.5
96	150	15.9	68.7	9.8	210.	10.0
97	150	30.2	36.2	24.6	799.	5.0
98	150	36.1	30.3	28.2	959.	4.5
99	150	64.7	16.9	32.1	1453.	3.5

In order to determine the relatonships of the fiber properties to the process and material parameters, the data of Table I were subjected to statistical analysis by multiple lnear regression. The regression equation obtained for fiber tenacity was as follows:

$$\text{Tenacity, g/d} = -8.47 + 2.00*SR + 0.491*IV + 0.0605*C*SR - 0.00623*T*SR - 0.0156*IV*SR - 0.00919*SR*SR$$

Where

SR is stretch ratio

IV is polymer intrinsic viscosity in decalin at 135° C., dl/g

C is polymer concentration in the gel, wt%

T is stretch temp. °C.

The statistics of the regression were:

F ratio (6,95)=118

significance level=99.9+%

standard error of estimate=3.0 g/d

A comparison between the observed tenacities and tenacities calculated from the regression equation is shown in FIG. 1.

FIGS. 2 and 3 present response surface contours for tenacity calculated from the regression equation on two important planes.

In the experiments of Examples 3–99, a correlation of modulus with spinning parameters was generally parallel to that of tenacity. A plot of fiber modulus versus tenacity is shown in FIG. 4.

It will be seen from the data, the regression equations and the plots of the calculated and observed results that the method of the invention enables substantial control to obtain desired fiber properties and that greater controlability and flexibility is obtained than by prior art methods.

Further, it should be noted that many of the fibers of these examples showed higher teancities and/or modulus values than had been obtained by prior art methods. In the prior art methods of Off. 30 04 699 and GB 2051667, all fibers prepared had tenacities less than 3.0 GPa (35 g/d) and moduli less than 100 GPa (1181 g/d). In the present instance, fiber examples Nos. 21, 67, 70, 73, 82, 84 and 88 exceeded both of these levels and other fiber examples surpassed on one or the other property.

In the prior art publications of Pennings and coworkers, all fibers (prepared discontinuously) had moduli less than 121 GPa (1372 g/d). In the present instance continuous fiber examples No. 70, 71, 73, 82, 83, 84, 88 and 99 surpassed this level.

The fiber of example 71 was further tested for resistance to creep at 23° C. under a sustained load of 10% of the breaking load. Creep is defined as follows:

$$\% \text{ Creep} = 100 \times (A(s,t) - B(s))/B(s)$$

where

B(s) is the length of the test section immediately after application of load

A(s,t) is the length of the test section at time t after application of load, s

A and B are both functions of the loads, while A is also a function of time t.

For comparison, a commercial nylon tire cord (6 denier, 9.6 g/d tenacity) and a polyethylene fiber prepared in accordance with Ser. No. 225,288, filed Jan. 15, 1981 by surface growth and subsequent hot stretching (10 denier, 41.5 g/d tenacity) were similarly tested for creep.

The results of these tests are presented in Table II.

TABLE II

CREEP RESISTANCE AT 23° C.
Load: 10% of Breaking Load

Time After Application of Load, Days	% Creep		
	Fiber of Example 71	Comparative Nylon Tire Cord	Surface Grown & Stretched Polyethylene
1	0.1	4.4	1.0
2	0.1	4.6	1.2
6	—	4.8	1.7
7	0.4	—	—
9	0.4	—	—
12	—	4.8	2.1
15	0.6	4.8	2.5
19	—	4.8	2.9
21	0.8	—	—
22	—	4.8	3.1
25	0.8	—	—
26	—	4.8	3.6
28	0.9	—	—
32	0.9	—	—
33	—	4.8	4.0
35	1.0	—	—
39	1.4	—	—
40	—	4.9	4.7
43	1.4	—	—
47	1.4	—	—
50	—	4.9	5.5
51	1.4	—	—
57	—	4.9	6.1
59	1.45	—	—

It will be seen that the fiber of example 71 showed about 1.4% creep in 50 days at 23° C. under the sustained load equal to 10% of the breaking load. By way of comparison, both the commercial nylon 6 tire cord and the surface grown polyethylene fiber showed about 5% creep under similar test conditions.

The melting temperatures and the porosities of the fibers of examples 64, 70 and 71 were determined. Melting temperatures were measured using a DuPont 990 differential scanning calorimeter. Samples were heated in an argon atmosphere at the rate of 10° C./min. Additionally, the melting temperature was determined for the starting polyethylene powder from which the fibers of examples 64, 70 and 71 were prepared.

Porosities of the fibers were determined by measurements of their densities using the density gradient technique and comparison with the density of a compression molded plaque prepared from the same initial polyethylene powder. (The density of the compression molded plaque was 960 kg/m^3).

Porosity was calculated as follows:

$$\%, \text{ Porosity} = \frac{960 - \text{fiber density, kg/m}^3}{960}$$

Results were as follows:

Sample	Melting Temp. °C.	Fiber Density, Kg/m^3	Porosity, %
Polyethylene powder	138	—	—
Fiber of Example 64	149	982	0
Fiber of Example 70	149	976	0
Fiber of Example 71	150	951	1

The particular level and combination of properties exhibited by the fiber of examples 64, 70 and 71, i.e., tenacity at least about 30 g/d, modulus in excess of 1000 g/d, and creep (at 23° C. and 10% of breaking load) less than 3% in 50 days, melting temperature of at least about 147° C. and porosity less than about 10% appears not to have been attained heretofore.

The following examples illustrate the effect of the second solvent upon fiber properties.

EXAMPLES 100–108

Fiber samples were prepared as described in Example 2, but with the following variations. The bottom discharge opening of the Helicone mixer was adapted to feed the polymer solution first to a gear pump and thence to a single hole conical spinning die. The cross-section of the spinning die tapered uniformly at a 7.5° angle from an entrance diameter of 10 mm to an exit diameter of 1 mm. The gear pump speed was set to deliver 5.84 cm^3/min of polymer solution to the die. The extruded solution filament was quenched to a gel state by passage through a water bath located at a distance of 20 cm below the spinning die. The gel filament was wound up continuously on bobbins at the rate of 7.3 meters/min.

The bobbins of gel fiber were immersed in several different solvents at room temperature to exchange with the paraffin oil as the liquid constituent of the gel. The solvents and their boiling points were:

Solvent	Boiling Point, °C.
diethyl ether	34.5
n-pentane	36.1
methylene chloride	39.8
trichlorotrifluoroethane	47.5
n-hexane	68.7
carbon tetrachloride	76.8
n-heptane	98.4
dioxane	101.4
toluene	110.6

The solvent exchanged gel fibers were air dried at room temperature. Drying of the gel fibers was accompanied in each case by substantial shrinkage of transverse dimensions. Surprisingly, it was observed that the shape and surface texture of the xerogel fibers departed progressively from a smooth cylindrical form in approximate proportion to the boiling point of the second solvent. Thus, the fiber from which diethyl ether had

been dried was substantially cylindrical whereas the fiber from which toluene had been dried was "C" shaped in cross-section.

The xerogel fibers prepared using TCTFE and n-hexane as second solvents were further compared by stretching each at 130° C., incrementally increasing stretch ratio until fiber breakage occurred. The tensile properties of the resulting fibers were determined as shown in Table III.

It will be seen that the xerogel fiber prepared using TCTFE as the second solvent could be stretched continuously to a stretch ratio of 49/1 and whereas the xerogel fiber prepared using n-hexane could be stretched continuously only to a stretch ratio of 33/1. At maximum stretch ratio, the stretched fiber prepared using TCTFE second solvent was of 39.8 g/d tenacity, 1580 g/d modulus. This compares to 32.0 g/d tenacity, 1140 g/d modulus obtained using n-hexane as the second solvent.

TABLE III

Properties of Xerogel Fibers Stretched at 130° C. Feed Speed: 2.0 cm/min.

Example	Second Solvent	Stretch Ratio	Tenacity g/d	Modulus g/d	Elong. %
100	TCTFE	16.0	23.3	740	5.0
101	TCTFE	21.8	29.4	850	4.5
102	TCTFE	32.1	35.9	1240	4.5
103	TCTFE	40.2	37.4	1540	3.9
104	TCTFE	49.3	39.8	1580	4.0
105	n-hexane	24.3	28.4	1080	4.8
106	n-hexane	26.5	29.9	920	5.0
107	n-hexane	32.0	31.9	1130	4.5
108	n-hexane	33.7	32.0	1140	4.5

EXAMPLE 110

Following the procedures of Examples 3-99, an 8 wt% solution of isotactic polypropylene of 12.8 intrinsic viscosity (in decalin at 135° C.), approximately 2.1×10^6 M.W. was prepared in paraffin oil at 200° C. A gel fiber was spun at 6.1 meters/min. The paraffin oil was solvent exchanged with TCTFE and the gel fiber dried at room temperature. The dried fiber was stretched 25/1 at a feed roll speed of 2 cm/min. Stretching was conducted in a continuous manner for one hour at 160° C.

Fiber properties were as follows:

denier—105

tenacity—9.6 g/d

modulus—164 g/d

elongation—11.5%

work-to-break—9280 in lbs/in^3 (64 MJ/m^3)

EXAMPLES 111-486

A series of xerogel fiber samples was prepared as in Example 2 but using a gear pump to control melt flow rate. Variations were introduced in the following material and process parameters:

a. polyethylene IV (molecular weight)
b. polymer gel concentration
c. die exit diameter
d. die included angle (conical orifice)
e. spinning temperature
f. melt flow rate
g. distance to quench
h. gel fiber take-up velocity
i. xerogel fiber denier

Each of the xerogel fiber samples prepared was stretched in a hot tube of 1.5 meter length blanketed with nitrogen and maintained at 100° C. at the fiber inlet and 140° C. at the fiber outlet. Fiber feed speed into the hot tube was 4 cm/min. (Under these conditions the actual fiber temperature was within 1° C. of the tube temperature at distances beyond 15 cm from the inlet). Each sample was stretched continuously at a series of increasing stretch ratios. The independent variables for these experiments are summarized below:

Polymer Intrinsic Viscosity (dL/g)

11.5—Examples 172-189, 237-241, 251-300, 339-371
15.5—Examples 111-126, 138-140, 167-171, 204-236, 242-243, 372-449, 457-459
17.7—Examples 127-137, 141-166, 190-203, 244-250, 301-338
20.9—Examples 450-456, 467-486

Gel Concentration	
5%	Examples 127-137, 141-149, 167-171, 190-203, 244-260, 274-276, 291-306, 339-371
6%	Examples 111-126, 138-140, 204-236, 242-243, 372-418, 431 ∝ 486
7%	Examples 150-166, 172-189, 237-241, 261-273, 277-290, 307-338

Die Diameter		
Inches	Millimeters	
0.04	1	Examples 167-171, 237-241, 244-260, 274-276, 282-290, 301-306, 317-338, 366-371 and 460-466
0.08	2	Examples 111-166, 172-236, 242, 243, 261-273, 277-281, 291-300, 307-316, 339-365, 372-459 and 467-486.

Die Angle (Degrees)	
0°	Examples 127-137, 141-149, 261-281, 307-316, 339-365, 419-430
7.5°	Examples 111-126, 138-140, 167-171, 204-243, 251-260, 301-306, 317-338, 372-418, 431-486
15°	Examples 150-166, 172-203, 244-250, 282-300, 366-371

Spinning Temperature	
180° C.	Examples 172-203, 237-241, 301-322, 339-371
200° C.	Examples 111-126, 138-140, 167-171, 204-236, 242-243, 372-486
220° C.	Examples 127-137, 141-166, 244-300, 323-338

Solution Flow Rate (cm^3/min)	
2.92 ± 0.02	Examples 116-122, 135-145, 150-152, 162-166, 172-173, 196-201, 214-222, 237, 240, 242-245, 251-255, 260-265, 277-284, 288-293, 301, 304-306, 310-312, 318-320, 347-360, 368-370, 372, 395-397, 401-407, 412-414, 419-424, 450-459, 467-481
4.37 ± 0.02	Examples 204-208, 230-236, 377-379, 408-411
5.85 ± 0.05	Examples 111-115, 123-134, 146-149, 153-161, 167-171, 180-195, 202-203, 209-213, 223-229, 238-239, 241, 256-259, 266-276, 285-287, 294-300, 302-303, 307-309, 315-317, 321-326, 335-338, 361-367, 371, 373-376, 392-394, 398-400, 415-418, 431-433, 482-486
6.07	Examples 339-346

-continued

8.76	Examples 380-391
8.88	Examples 246-250
11.71 ± 0.03	Examples 434-437, 445-449
17.29	Examples 438-440

Distance to Quench		
Inches	Millimeters	Examples
5.5	140	116-126
6.0	152	127-137, 158-166, 172-173, 183-198, 222-229, 240-243, 246-259, 282-286, 293-296, 301, 302, 323-330, 366-368, 398-407, 419-430
6.5	165	268-273, 277-281
7.7	196	167-171
13.0	330	450-453
14.5	368	377-391
15.0	381	230-236, 408-411, 431-449, 454-456, 467-486
22.5	572	307-312, 339-349
23.6	600	111-115, 138-140
24.0	610	141-157, 174-182, 199-203, 209-221, 244-245, 287-292, 297-300, 303-306, 319-322, 331-338, 372, 392-394, 412-418, 460-466

Under all of the varied conditions, the take-up velocity varied from 90-1621 cm/min, the xerogel fiber denier from 98-1613, the stretch ratio from 5-174, the tenacity from 9-45 g/denier, the tensile modulus from 218-1700 g/denier and the elongation from 2.5-29.4%.

The results of each Example producing a fiber of at least 30 g/denier (2.5 GPa) tenacity or at least 1000 g/denier (85 GPa) modulus are displayed in Table IV.

TABLE IV

	Stretched Fiber Properties				
Example	Xerogel Fiber Denier	Stretch Ratio	Tenacity g/den	Modulus g/den	% Elong
113	1599.	50.	31.	1092.	4.0
114	1599.	57.	34.	1356.	3.6
115	1599.	72.	37.	1490.	3.5
119	1837.	63.	35.	1257.	4.2
122	1289.	37.	32.	988.	4.5
126	440.	41.	31.	1051.	4.5
128	1260.	28.	31.	816.	5.5
130	1260.	33.	33.	981.	4.5
131	1260.	43.	35.	1179.	4.0
132	1260.	40.	37.	1261.	4.5
133	1260.	39.	30.	983.	4.0
134	1260.	53.	36.	1313.	4.0
135	282.	26.	29.	1062.	3.5
136	282.	26.	30.	1034.	3.5
137	282.	37.	30.	1261.	3.5
140	168.	23.	26.	1041.	3.5
145	568.	40.	30.	1157.	4.0
146	231.	21.	32.	763.	4.0
147	231.	25.	36.	1175.	4.2
148	231.	22.	33.	1131.	4.0
149	231.	19.	31.	1090.	4.0
151	273.	31.	28.	1117.	3.5
157	1444.	64.	29.	1182.	3.0
160	408.	35.	30.	1124.	4.0
164	1385.	36.	32.	1210.	4.0
166	1385.	39.	33.	1168.	4.0
168	344.	26.	30.	721.	5.0
169	344.	40.	32.	1188.	4.0
170	344.	26.	30.	1060.	4.0
171	344.	29.	31.	1172.	4.0
179	1017.	68.	29.	1179.	4.0
182	352.	65.	33.	1146.	3.7
189	1958.	44.	27.	1050.	3.5
195	885.	59.	31.	1150.	4.0
201	496.	33.	29.	1082.	4.0
206	846.	37.	31.	955.	4.5
208	846.	63.	35.	1259.	3.5

TABLE IV-continued

	Stretched Fiber Properties				
Example	Xerogel Fiber Denier	Stretch Ratio	Tenacity g/den	Modulus g/den	% Elong
212	368.	55.	39.	1428.	4.5
213	368.	49.	35.	1311.	4.0
220	1200.	81.	34.	1069.	4.0
221	1200.	60.	30.	1001.	4.0
227	1607.	42.	30.	1050.	4.0
228	1607.	47.	30.	1114.	3.5
229	1607.	53.	35.	1216.	4.0
233	1060.	34.	30.	914.	4.5
235	1060.	50.	37.	1279.	4.1
236	1060.	74.	45.	1541.	4.0
245	183.	23.	26.	1014.	4.0
247	247.	16.	30.	1005.	4.5
248	247.	10.	30.	1100.	4.0
249	247.	11.	31.	1132.	4.0
250	247.	19.	37.	1465.	3.8
251	165.	34.	31.	1032.	4.5
252	165.	33.	31.	998.	4.5
254	165.	41.	31.	1116.	4.0
255	165.	40.	29.	1115.	4.0
272	1200.	41.	24.	1122.	3.0
273	1200.	64.	27.	1261.	2.5
274	154.	27.	30.	854.	4.5
275	154.	44.	32.	1063.	4.5
276	154.	38.	30.	1054.	4.0
280	291.	39.	30.	978.	4.0
281	291.	43.	29.	1072.	4.0
284	254.	30.	32.	1099.	4.5
308	985.	27.	30.	900.	4.3
309	985.	34.	35.	1210.	3.8
311	306.	30.	31.	990.	4.4
312	306.	30.	32.	1045.	4.0
314	1234.	45.	37.	1320.	4.0
315	344.	25.	30.	970.	4.0
317	254.	29.	32.	1270.	3.5
320	190.	29.	30.	1060.	4.0
322	307.	25.	29.	1030.	4.0
323	340.	25.	34.	1293.	4.1
324	340.	23.	33.	996.	4.4
325	340.	30.	37.	1241.	4.1
326	340.	35.	39.	1480.	3.7
327	373.	24.	30.	920.	4.5
328	373.	27.	34.	1080.	4.5
329	373.	30.	36.	1349.	4.0
330	373.	35.	37.	1377.	3.9
332	218.	34.	35.	1320.	3.9
333	218.	30.	37.	1364.	4.0
334	218.	30.	31.	1172.	3.9
335	326.	26.	37.	1260.	4.5
336	326.	30.	39.	1387.	4.2
337	326.	42.	42.	1454.	4.0
338	326.	42.	37.	1440.	3.9
339	349.	55.	29.	1330.	3.3
345	349.	31.	29.	1007.	4.5
346	349.	51.	34.	1165.	4.3
357	772.	45.	31.	990.	4.4
358	772.	51.	27.	1356.	3.0
359	772.	58.	32.	1240.	3.7
360	772.	59.	33.	1223.	3.8
364	293.	47.	38.	1407.	4.5
375	1613.	50.	30.	960.	4.1
379	791.	46.	32.	1110.	3.9
382	1056.	68.	34.	1280.	3.7
383	921.	51.	31.	1090.	4.0
386	1057.	89.	34.	1250.	3.8
387	984.	59.	33.	1010.	4.3
394	230.	29.	31.	982.	4.3
400	427.	32.	30.	970.	4.1
405	1585.	39.	33.	1124.	3.6
407	1585.	174.	32.	1040.	4.0
418	1370.	51.	33.	1160.	3.7
419	344.	23.	30.	1170.	3.8
421	1193.	30.	31.	880.	4.6
422	1193.	39.	35.	1220.	3.9
423	1193.	51.	34.	1310.	3.4
424	1193.	50.	36.	1390.	3.6
426	1315.	32.	30.	860.	4.4
427	1315.	42.	33.	1160.	3.9

TABLE IV-continued

Example	Xerogel Fiber Denier	Stretch Ratio	Tenacity g/den	Modulus g/den	% Elong
	Stretched Fiber Properties				
428	1315.	46.	34.	1170.	3.8
429	395.	19.	35.	840.	4.5
430	395.	25.	31.	1100.	3.9
435	1455.	36.	31.	920.	4.3
436	1455.	43.	31.	1120.	3.6
437	1455.	51.	33.	1060.	3.3
440	1316.	37.	32.	1130.	4.0
441	453.	31.	32.	990.	4.7
442	453.	49.	39.	1320.	4.4
443	453.	34.	33.	1060.	4.4
444	453.	55.	36.	1410.	3.6
446	402.	28.	30.	1107.	4.0
447	402.	22.	30.	870.	5.0
448	402.	34.	36.	1175.	4.3
449	402.	38.	37.	1256.	4.3
451	461.	33.	33.	1070.	4.4
452	461.	38.	35.	1130.	4.1
453	461.	40.	35.	1220.	3.7
454	64.	14.	34.	1080.	4.7
455	64.	17.	35.	1263.	3.4
456	64.	26.	40.	1453.	3.8
460	268.	32.	35.	1220.	4.3
462	268.	29.	34.	1100.	4.2
463	268.	32.	34.	1110.	4.1
464	268.	43.	40.	1390.	3.9
465	420.	53.	41.	1550.	3.7
466	420.	27.	31.	1010.	4.0
467	371.	24.	31.	960.	4.4
468	371.	63.	45.	1560.	3.9
470	1254.	40.	35.	1100.	4.1
471	1254.	43.	37.	1190.	4.0
472	1254.	45.	38.	1320.	4.0
473	1254.	66.	39.	1600.	3.5
474	210.	44.	43.	1700.	3.5
475	210.	21.	34.	1170.	4.0
476	210.	27.	38.	1420.	3.6
479	1227.	50.	34.	1180.	4.1
480	1227.	48.	33.	1140.	4.1
481	1227.	44.	35.	1230.	4.1
483	1294.	29.	31.	1000.	4.3
484	1294.	42.	36.	1350.	3.7
485	340.	26.	32.	1160.	3.8
486	340.	18.	27.	1020.	4.1

In order to determine the relationships of the fiber properties to the process and material parameters, all of the data from Example 111–486, including those Examples listed in Table IV, were subjected to statistical analysis by multiple linear regression. The regression equation obtained for fiber tenacity was as follows:

$$\text{Tenacity, g/d} = 11.88 + 2.221IV' + 1.147C' + 1.948TM' + 0.822Q' - 1.167L' - 2.438DO' + 0.532SR - 0.7261V'DA' + 1.399IV'TM' + 0.534IV'L' + 0.046IV'SR - 0.754C'DA' - 0.391C'Q' - 0.419C'DO' - 1.327D'TM' + 0.366D'L' - 0.577DA'TM' - 0.790DA'Q' - 0.034DA'SR - 0.049TM'SR + 0.809Q'L' - 0.313Q'DO' - 0.334(IV')^2 + 0.115(L')^2 + 0.564(DO')^2 - 0.00237(SR)^2$$

where:

IV'=(polymer IV, dL/g—14.4)/3.1

C'=Gel concentration, %—6

TM'=(spinning temp. °C.—200)/20

Q'=(spin flow rate, cc/min—4.38)/1.46

L'=(distance to quench, in—15)/9

DO'=1.4427 log (xerogel fiber denier/500)

SR=stretch ratio (xerogel fiber denier/stretched fiber denier)

DA'=(die angle, °—7.5)/7.5

D'=(die exit diameter, inches—0.06)/0.02

The statistics of the reggression were;

F ratio (26, 346)=69

Significance Level=99.9+%

Standard error of estimate=2.6 g/denier

In the vicinity of the center of the experimental space these effects may be summarized by considering the magnitude of change in the factor which is required to increase tenacity of 1 g/d. This is given below.

Factor		Factor Change Required to Increase Tenacity By 1 g/denier
IV	+1	dL/g
Conc.	+1	wt %
Spin Temp.	+10	°C.
Spin Rate	±(saddle)	cc/min
Die Diam.	−0.010	inches
Die Angle	−2	degrees
Dist. to Quench	−4	inches
Xerogel Fiber Denier	−25	
Stretch Ratio	+2/1	

High fiber tenacity was favored by increasing polymer IV, increasing gel concentration, increasing spinning temperature, decreasing die diameter, decreasing distance to quench, decreasing xerogel fiber diameter, increasing stretch ratio and 0° die angle (straight capillary).

It will be seen that the method of the invention enables substantial control to obtain desired fiber properties and that greater controlability and flexability is obtained than by prior art methods.

In these experiments, the effects of process parameters upon fiber modulus generally paralled the effects of these variables upon tenacity. Fiber modulus was correlated with tenacity as follows

modulus, g/d=42(tenacity, g/d)−258

Significance of the correlation between modulus and tenacity was 99.99+%. Standard error of the estimate of modulus was 107 g/d.

It should be noted that many of the fibers of these examples show higher tenacities and/or higher modulus than had seen obtained by prior art methods.

The densities and porosities of several of the xerogel and stretched fibers were determined.

Example	Xerogel fiber Density kg/m³	Xerogel fiber % Porosity	Stretched fiber Density, kg/m³	Stretched fiber % Porosity
115	934	2.7	—	—
122	958	0.2	0.965	0
126	958	0.2	—	—
182	906	5.6	940	2.1

The porosities of these samples were substantially lower than in the prior art methods cited earlier.

EXAMPLES 487–583

In the following examples of multi-filament spinning and stretching, polymer solutions were prepared as in Example 2. The solutions were spun through a 16 hole spinning die using a gear pump to control solution flow rate. The aperatures of the spinning die were straight capillaries of length-to-diameter ratio of 25/1. Each capillary was preceded by a conical entry region of 60° included angle.

The multi-filament solution yarns were quenched to a gel state by passing through a water bath located at a short distance below the spinning die. The gel yarns were wound up on perforated dye tubes.

EXAMPLES 487–495

ONE STAGE "DRY STRETCHING" OF MULTI-FILAMENT YARN

The wound tubes of gel yarn were extracted with TCTFE in a large Sohxlet apparatus to exchange this solvent for paraffin oil as the liquid constituent of the gel. The gel fiber was unwound from the tubes and the TCTFE solvent was evaporated at room temperature.

The dried xerogel yarns were stretched by passing the yarn over a slow speed feed godet and idler roll through a hot tube blanketed with nitrogen, onto a second godet and idler roller driven at a higher speed. The stretched yarn was collected on a winder.

It was noted that some stretching of the yarn (approximately 2/1) occurred as it departed the feed godet and before it entered the hot tube. The overall stretch ratio, i.e., the ratio of the surface speeds of the godets, is given below.

In examples 487–495, the diameter of each hole of the 16 filament spinning die was 0.040 inch one millimeter) the spinning temperature was 220° C., the stretch temperature (in the hot tube) was 140° C. and the feed roll speed during stretching was 4 cm/min. In examples 487–490 the polymer IV was 17.5 and the gel concentration was 7 weight %. In examples 491–495 the polymer IV was 22.6. The gel concentration was 9 weight % in example 491, 8 weight % in examples 492–493 and 6 weight % in examples 494 and 495. The distance from the die face to the quench bath was 3 inches (7.52 cm) in examples 487, 488, 494 and 495 and 6 inches (15.2 cm) in examples 490–493. The other spinning conditions and the properties of the final yarns were as follows:

		Yarn Properties					
Ex. No.	Spin Rate cc/min-fil	Gel Fiber Take-up Speed cc/min	SR	Denier	Ten g/d	Mod g/d	% Elong
487	1.67	1176	35	41	36	1570	3.3
488	2.86	491	25	136	27	1098	3.7
489	2.02	337	25	132	29	1062	3.6
490	2.02	337	30	126	31	1275	3.5
491	1.98	162	25	151	33	1604	3.0
492	1.94	225	25	227	29	1231	3.3
493	1.94	225	30	143	34	1406	3.3
494	1.99	303	30	129	34	1319	3.4
495	1.99	303	35	112	35	1499	3.2

EXAMPLES 496–501

ONE STAGE "WET STRETCHING" OF MULTI-FILAMENT YARN

The wound gel yarns still containing the paraffin oil were stretched by passing the yarn over a slow speed feed godet and idler roll through a hot tube blanketed with nitrogen onto a second godet and idler roll driven at high speed. It was noted that some stretching of the yarn (approximately 2/1) occurred as it departed the feed godet and before it entered the hot tube. The overall stretch ratio, i.e., the ratio of the surface speeds of the godets is given below. The stretching caused essentially no evaporation of the paraffin oil (the vapor pressure of the paraffin oil is about 0.001 atmospheres at 149° C.). However, about half of the paraffin oil content of the gel yarns was exuded during stretching. The stretched gel yarns were extracted with TCTFE in a Sohxlet apparatus, then unwound and dried at room temperature.

In each of the examples 496–501 the spinning temperatures was 220° C., the gel concentration was 6 weight % the distance from the spinning die to the water quench was 3 inches (7.6 cm).

In examples 496 and 499–501 the diameter of each hole of the spinning die was 0.040 inches (0.1 cm). In examples 497 and 498 the hole diameters were 0.030 inches (0.075 cm). In examples 496 and 494–501 the polymer IV was 17.5. In examples 497 and 498 the polymer IV was 22.6. The other spinning conditions and properties of the final yarns were as follows:

Ex. No.	Spinning Rate cc/min-fil	Gel Fiber Take-up Speed cm/min	Stretch Temp	Stretch Ratio	Denier
496	2.02	313	140	22	206
497	1.00	310	140	12.5	136
498	1.00	310	140	15	94
499	2.02	313	120	20	215
500	2.02	313	120	22.5	192
501	2.02	313	120	20	203

Ex. No.	Tenacity g/d	Modulus g/d	% Elong
496	25	1022	3.7
497	28	1041	3.6
498	32	1389	2.8
499	30	1108	4.5
500	30	1163	4.2
501	27	1008	4.2

EXAMPLES 502–533

In the following examples a comparison is made between alternative two stage modes of stretching the same initial batch of yarn. All stretching was done in a hot tube blanketed with nitrogen.

EXAMPLE 502

GEL YARN PREPARATION

The gel yarn was prepared from a 6 weight % solution of 22.6 IV polyethylene as in example 2. The yarn was spun using a 16 hole×0.030 inch (0.075 cm) die. Spinning temperature was 220° C. Spin rate was 1 cm^3/min-fil. Distance from the die face to the quench bath was 3 inches (7.6 cm). Take-up speed was 308 cm/min. Nine rolls of 16 filament gel yarn was prepared.

EXAMPLES 503–576

"WET-WET" STRETCHING

In this mode the gel yarn containing the paraffin oil was stretched twice. In the first stage, three of the rolls of 16 filament gel yarns described in example 502 above were combined and stretched together to prepare a 48 filament stretched gel yarn. The first stage stretching conditions were: Stretch temperature 120° C., feed speed 35 cm/min, stretch ratio 12/1. A small sample of the first stage stretched gel yarn was at this point extracted with TCTFE, dried and tested for tensile properties. The results are given below as example 503.

The remainder of the first stage stretched gel yarn was restretched at 1 m/min feed speed. Other second stage stretching conditions and physical properties of the stretched yarns are given below.

Ex. No.	2nd Stage Stretch Temp - °C.	2nd Stage Stretch Ratio	Denier	Tenacity g/d
503	—	—	504	22
504	130	1.5	320	28
505	130	1.75	284	29
506	130	2.0	242	33
507	140	1.5	303	31
508	140	1.75	285	32
509	140	2.25	222	31
510	145	1.75	285	31
511	145	2.0	226	32
512	145	2.25	205	31
513	150	1.5	310	28
514	150	1.7	282	28
515	150	2.0	225	33
516	150	2.25	212	31

Ex. No.	Modulus g/d	% Elong	Melting* Temp, °C.
503	614	5.5	147
504	1259	2.9	—
505	1396	2.6	150, 157
506	1423	2.8	—
507	1280	3.1	—
508	1367	3.0	149, 155
509	1577	2.6	—
510	1357	3.0	—
511	1615	2.7	—
512	1583	2.5	151, 156
513	1046	3.0	—
514	1254	2.9	—
515	1436	2.9	—
516	1621	2.6	152, 160

*The unstretched xerogel melted at 138° C.

The density of the fiber of example 515 was determined to be 980 kg/m^3. The density of the fiber was therefore higher than the density of a compression molded plaque and the porosity was essentially zero.

EXAMPLES 517–522

"WET-DRY" STRETCHING

In this mode the gel yarn was stretched once then extracted with TCTFE, dried and stretched again.

In the first stage, three of the rolls of 16 filament gel yarn described in Example 502 were combined and stretched together to prepare a 48 filament stretched gel yarn. The first stage stretching conditions were: stretch temperature 120° C., feed speed 35 cm/min, stretch ratio 12/1.

The first stage stretchd gel yarn was extracted with TCTFE in a Sohxlet apparatus, rewound and air dried at room temperature, then subjected to a second stage of stretching in the dry state at a feed speed of 1 m/min. Other second stage stretching conditions and physical properties of the stretching yarn are given below..

Ex. ample	2nd Stage Stretch Temp, °C.	2nd Stage Stretch Ratio	Den- ier	Ten g/d	Mod g/d	% Elong.	Melt Temp, °C.
517	130	1.25	390	22	1193	3.0	—
518	130	1.5	332	26	1279	2.9	150, 157
519	140	1.5	328	26	1291	3.0	—
520	140	1.75	303	27	1239	2.7	150, 159
521	150	1.75	292	31	1427	3.0	—
522	150	2.0	246	31	1632	2.6	152, 158

EXAMPLES 523–533

"DRY-DRY" STRETCHING

In this mode the gel yarn described in example 502 was extracted with TCTFE, dried, then stretched in two stages. In the first stage, three of the rolls of 16 filament yarn were combined and stretched together to prepare a 48 filament stretched xerogel yarn. The first stage stretching conditions were: stretch temperature 120° C., feed speed 35 cm/min., stretch ratio 10/1. The properties of the first stage stretched xerogel yarn are given as example 523 below. In the second stretch stage the feed speed was 1 m/min. Other second stage stretching conditions and physical properties of the stretched yarns are given below.

Ex- am- ple	Stretch Temp, °C.	SR	Denier	Ten g/d	Mod g/d	% Elong.	Melt Temp, °C.
523	—	—	392	21	564	4.3	146, 153
524	130	1.5	387	24	915	3.1	—
525	130	1.75	325	23	1048	2.4	150, 158
526	140	1.5	306	28	1158	2.9	—
527	140	1.75	311	28	1129	2.9	—
528	140	2.0	286	24	1217	2.3	150, 157
529	150	1.5	366	26	917	3.3	—
530	150	1.75	300	28	1170	3.0	—
531	150	2.0	273	31	1338	3.8	—
532	150	2.25	200	32	1410	2.2	—
533	150	2.5	216	33	1514	2.5	152, 156

The density of the fiber of example 529 was determined to be 940 Kg/m^3. The porosity of the fiber was therefore about 2%.

EXAMPLES 534–542

MULTI-STAGE STRETCHING OF MULTI-FILAMENT YARN

In the following examples a comparison is made between two elevated temperatures stretches and a three stage stretch with the first stage at room temperature. The same initial batch of polymer solution was used in these examples.

EXAMPLE 534

UNSTRETCHED GEL YARN PREPARATION

A 6 weight % solution of 22.6 IV polyethylene yarn was prepared as in example 2. A 16 filament yarn was spun and wound as in example 502.

EXAMPLE 535

PREPARATION OF GEL YARN STRETCHED AT ROOM TEMPERATURE

The unstretched gel yarn prepared as in example 534 was led continuously from a first godet which set the spinning take-up speed to a second godet operating at a surface speed of 616 cm/min. In examples 540–542 only, the as-spun gel fiber was stretched 2/1 at room temperature in-line with spinning. The once stretched gel fiber was wound on tubes.

EXAMPLES 536-542

The 16 filament gel yarns prepared in examples 534 and 535 were stretched twice at elevated temperature. In the first of such operations the gel yarns were fed at 35 cm/min to a hot tube blanketed with nitrogen and maintained at 120° C. In the second stage of elevated temperature stretching the gel yarns were fed at 1 m/min and were stretching at 150° C. Other stretching conditions and yarn properties are given below.

Example	SR RT	SR 120° C.	SR 150° C.	Total SR	Denier	Ten g/den	Mod g/den	Elong
536	—	8.3	2.25	18.7	128	23	1510	2.6
537	—	8.3	2.5	20.8	116	30	1630	3.0
538	—	8.3	2.75	22.8	108	30	1750	2.7
539	—	8.3	3.0	24.9	107	31	1713	2.6
540	2	6.8	2.0	27.2	95	30	1742	2.5
541	2	6.8	2.25	30.6	84	34	1911	2.5
542	2	6.8	2.5	34	75	32	1891	2.2

EXAMPLES 543-551

POLYETHYLENE YARNS OF EXTREME MODULUS

The highest experimental value reported for the modulus of a polyethylene fiber appears to be by P. J. Barham and A. Keller, J. Poly. Sci., Polymer Letters ed. 17, 591 (1979). The measurement 140 GPa (1587 g/d) was made by a dynamic method at 2.5 Hz and 0.06% strain and is expected to be higher than would be a similar measurement made by A.S.T.M. Method D2101 "Tensile Properties of Single Man Made Fibers Taken from Yarns and Tows" or by A.S.T.M. Method D2256 "Breaking Load (Stength) and Elongation of Yarn by the Single Strand Method." The latter methods were used in obtaining the data reported here.

The following examples illustrate the preparation of novel polyethylene yarns of modulus exceeding 1600 g/d and in some cases of modulus exceeding 2000 g/d. Such polyethylene fibers and yarns were heretofore unknown. In the following examples all yarns were made from a 22.6 IV polyethylene, 6 weight % solution prepared as in example 2 and spun in example 502. All yarns were stretched in two stages. The first stage stretch was at a temperature of 120° C. The second stage stretch was at a temperature of 150° C. Several 16 filament yarn ends may have been combined during stretching. Stretching conditions and yarn properties are given below.

Example	Feed-1 cm/min	SR-1	Feed-2 cm/min	SR-2	Fils	Ten g/den	Mod g/den	Elong
			Wet - Wet					
543	25	15	100	2.25	48	39	1843	2.9
544	35	12.5	100	2.5	64	31	1952	2.6
545	35	10.5	100	2.75	48	31	1789	2.4
546	100	6.4	200	2.85	48	27	1662	2.5
			Wet - Dry					
547	25	15	100	2.0	48	36	2109	2.5
548	25	15	100	2.0	48	32	2305	2.5
549	25	15	100	2.0	48	30	2259	2.3
550	25	15	100	1.87	48	35	2030	2.7
551	25	15	100	1.95	16	35	1953	3.0

The yarns of examples 548 and 550 were characterized by differential scanning calorimetry and density measurement. The results, displayed below, indicate two distinct peaks at the melting points indicated, quite unlike the broad single peak at 145.5° C. or less reported by Smith and Lemstra in J. Mat. Sci., vol 15, 505 (1980).

Example	Melt Temp(s)	Density	% Porosity
548	147, 155° C.	977 kg/m^3	0
550	149, 156° C.	981 kg/m^3	0

EXAMPLES 552-558

POLYPROPYLENE YARNS OF EXTREME MODULUS

The highest reported experimental value for the modulus of a polypropylene material (fiber or other form) appears to be by T. Williams, J. Mat. Sci. 8, 59 (1973). Their value on a solid state extruded billet was 16.7 GPa (210 g/d). The following examples illustrate the preparation of novel polypropylene continuous fibers with modulus exceeding 220 g/d and in some cases of modulus exceeding 250 g/d.

In the following examples all fibers were made from an 18 IV polypropylene, 6 weight % solution in paraffin oil prepared as in example 2. In Examples 552-556, the fibers were spun with a single hole conical die of 0.040" (0.1 cm) exit diameter and 7.5% angle. Melt temperature was 220° C. A melt pump was used to control solution flow rate at 2.92 cm^3/min. Distance from the die face to the water quench was 3 inches (7.6 cm). The gel fibers were one stage wet stretched at 25 cm/min feed roll speed into a 1.5 m hot tube blanketed with nitrogen. The stretched fibers were extracted in TCTFE and air dried. Other spinning and stretching conditions as well as fiber properties are given below.

Example	Gel Fiber Take-up Speed	Stretch Temp °C.	SR	Denier	Ten g/d	Mod g/d	Elong
552	432	139	10	33	13.0	298	15.8
553	432	138	10	34	13.0	259	18.3

-continued

Example	Gel Fiber Take-up Speed	Stretch Temp °C.	SR	Denier	Ten g/d	Mod g/d	Elong
554	317	140	5	45	11.2	262	19.9
555	317	140	10	51	11.0	220	19.6
556	317	150	10	61	8.8	220	29.8

The fiber of example 556 determined by differential scanning calorimetry to have a first melting temperature of 170°–171° C. with higher order melting temperatures of 173° C., 179° C. and 185° C. This compares with the 166° C. melting point of the initial polymer. The moduli of these fibers substantially exceed the highest previously reported values.

In Examples 557 and 558, the yarns were spun with a 16 hole×0.040 inch (1 mm) capillary die. The solution temperature was 223° C., and the spinning rate was 2.5 cm^3/min-filament. The distance from the die face to the water quench bath was 3 inches (7.6 cm). Take-up speed was 430 cm/min. The gel yarns were "wet-wet" stretched in two stages. The first stage stretching was at 140° C. at a feed speed of 35 cm/min. The second stage stretching was at a temperature of 169° C., a feed speed of 100 cm/min and a stretch ratio of 1.25/1. Other stretching conditions as well as fiber properties are given below.

Example	SR-1	Denier	Ten g/den	Mod g/den	% Elong.
557	9.5	477	10	368	6.8
558	9.0	405	10	376	5.7

The moduli of these yarns very substantially exceed the highest previously reported values.

We claim:

1. A stretched polyethylene fiber of substantially indefinite length being of weight average molecular weight at least about 500,000 and having a tenacity of at least about 20 g/denier, a tensile modulus at least about 500 g/denier, a creep value no more than about 5% (when measured at 10% of breaking load for 50 days at 23° C.), a porosity less than about 10% and a main melting temperature of at least about 147° C. (measured at 10° C./minute heating rate by differential scanning calorimetry).

2. The stretched polyethylene fiber of claim 1 having a tenacity of at least about 30 g/denier and a tensile modulus of at least about 1000 g/denier.

3. The stretched polyethylene fiber of claim 2 having a tensile modulus of at least about 1600 g/denier.

4. The stretched polyethylene fiber of claim 2 having a tensile modulus of at least about 2000 g/denier.

5. The stretched polyethylene fiber of claim 1 or 2 having a main melting temperature at least about 149° C. (measured at 10° C./minute heating rate by differential scanning calorimetry).

6. The stretched polyethylene fiber of claim 1 or 2 or 3 or 4 having a main melting temperature of at least about 149° C. (measured at 10°/minute heating rate by differential scanning calorimetry).

7. The stretched polyethylene fiber of claim 1 or 2 or 3 or 4 being of weight average molecular weight of at least about 1,000,000.

8. The stretched polyethylene fiber of claim 1 or 2 or 3 or 4 being of weight average molecular weight between about 2,000,000 and about 8,000,000.

9. A stretched polyethylene fiber of substantially indefinite length being of weight average molecular weight of at least about 1,000,000 and having a tensile modulus of at least about 1600 g/denier, a main melting temperature of at least about 147° C. (measured at 10° C./minute heating rate by differential scanning calorimetry) and an elongation-to-break of not more than 5%.

10. The stretched polyethylene fiber of claim 9 being of weight average molecular weight between about 2,000,000 and about 8,000,000.

11. The stretched polyethylene fiber of claim 9 or 10 having a main melting temperature of at least about 149° C. (measured at 10° C./minute heating rate by differential scanning calorimetry).

12. The stretched polyethylene fiber of claim 9 or 10 having a tensile modulus of at least about 2000 g/denier.

13. A stretched polypropylene fiber of substantially indefinite length being of weight average molecular weight of at least about 750,000 and having a tenacity of at least about 8 g/denier, a tensile modulus of at least about 160 g/denier and a main melting temperature of at least about 168° C. (measured at 10° C./minute heating rate by differential scanning calorimetry).

14. The stretched polypropylene fiber of claim 13 having a tenacity of at least about 11 g/denier.

15. The stretched polypropylene fiber of claim 13 having a tenacity of at least about 13 g/denier.

16. The stretched polypropylene fiber of claim 13 having a tensile modulus of at least about 200 g/denier.

17. The stretched polypropylene fiber of claim 13 having a tensile modulus of at least about 220 g/denier.

18. The stretched polypropylene fiber of claim 13 or 14 or 15 or 16 or 17 being of weight average molecular weight at least about 1,000,000.

19. The stretched polypropylene fiber of claim 13 or 14 or 15 or 16 or 17 being of weight average molecular weight between about 2,000,000 and about 8,000,000.

* * * * *

BROOKSTEIN DECLARATION EXHIBIT 12

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TEXTILE SCIENCE AND TECHNOLOGY 6

PRODUCTION AND APPLICATIONS OF POLYPROPYLENE TEXTILES

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2.3.3 *Important physical and mechanical properties of polypropylene fibres*

2.3.3.1 Specific volume, density, covering power, appearance and fineness

Polypropylene fibres made of isotactic polymer have a density in the range of 0.90–0.91 g cm^{-3}; the fibres are therefore lighter than water. They have practically the highest specific volume, thus giving also the highest covering power of all common textile fibres.

Some characteristics in this respect for most commercial textile fibres are compared in Table 6. The cross-sectional area given here has been calculated under the presumption that all the fibres have a 0.11 tex fineness and a circular cross-section.

Table 6 — Specific parameters of fibres

Fibre	Density [g cm^{-3}]	Specific volume [cm^3 g^{-1}]	Cross-sectional area [μm^2 tex^{-1}]	[%]
Polypropylene	0.92	1.09	1087	100
Nylon	1.14	0.88	877	81
Acrylic	1.18	0.85	847	78
Polyester	1.38	0.72	725	67
Wool	1.32	0.76	756	70
Cotton	1.50	0.67	667	65
Viscose rayon	1.52	0.66	658	60

From the table it can be derived that the polypropylene fibre gives better cover than nylon, acrylic and polyester fibres by 19, 22 and 33 %, respectively. And when compared with viscose rayon, this difference reaches as much as 40 % in favour of polypropylene.

In other words, when different fibres of the same covering power and, necessarily, of different fineness are used in making comparable

fabrics, the fabrics in theory are lighter or heavier according to what fibres are constituting them.

To give an example on the cover equivalence, some fibres of the same covering power as a 0.33 tex polypropylene fibre are ranked as follows:

0.42 tex nylon fibre
0.43 tex acrylic fibre
0.46 tex triacetate or polyvinylalcohol fibre
0.50 tex polyester or polyvinylchloride fibre
0.55 tex viscose rayon fibre
0.78 tex polytetrafluoroethylene fibre
0.90 tex glass fibre

It follows from the above comparison that, in theory, a knitted or woven fabric made of glass fibre is about three times heavier than that made of polypropylene fibre provided, of course, that the fabrics are of equal construction in order to be comparable.

The given data can be used as a guide only with simple fibres, monofilaments and tapes; with multifilaments and spun yarns a correction should be taken into account due to the porosity.

Appearance and fineness of polypropylene fibres

Polypropylene fibres are white and can vary from lustrous and semi-translucent to dull and opaque in appearance, depending on the amount of delustrant added to the polymer. They can also be dope-dyed to various shades of high fastness.

The fibres have a waxy-to-soapy handle and a smooth, uniform surface, and are mostly circular in cross-section, though some other cross-sectional shapes are also available. They are produced in the fineness ranging from 0.11 to 2.2 tex.

2.3.3.2 Tensile properties

Polypropylene fibres are produced in different forms, such as staple, monofilament, multifilament and low- and high-denier tows, and are modified to suit a particular textile or technical end-use. From the viewpoint of tensile properties they can be divided into the following three groups:

Fibres with a medium tenacity of 400 – 600 mN tex^{-1}

These are the main fibres produced and used most commonly, both as staple and as filament.

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Fibres with a high tenacity of 900 – 1300 mN tex^{-1}

The fibres are intended for special, technical and military uses and are produced as staple, filament and low-denier tow.

Fibres with a low tenacity of 200 – 300 mN tex^{-1}

Staple fibres of this kind are used to advantage in the production of carpet yarns, bestowing on the yarns a high resiliency at the expense of strength. Nevertheless, the strength of the yarns is still sufficient for the purpose. They are commercially produced under such trade names as Meraklon SR and Meraklon DR.

The loop and knot strengths are lower by some 10 – 20 % than the tensile strength. The moisture regain at 21 °C and 65 % R.H. is below 0.1 % and this is why the fibre tenacity remains practically unaltered even after the fibres have been wetted, boiled or steamed for a long time.

Elongation

On average, the elongation varies according to the fibre form as follows:

- monofilament 15 – 30 %
- multifilament 20 – 80 %
- normal-tenacity staple 20 – 35 %
- low-tenacity staple 70 – 100 %
- Meraklon SR 70 – 90 %
- Meraklon DR 70 – 100 %

Load-elongation curves, obtained under standard conditions, for some fibres are shown in Fig. 24. From it a comparison can be made of some kinds of polypropylene fibres including filament, standard staple (Meraklon S, cotton-type) and low-tenacity staple (Meraklon SR, carpet-type), and other commercially important fibres.

2.3.3.3 Elastic properties

Elastic properties of polypropylene fibres can vary along with other mechanical properties in a wide range of values. They depend on the type of polymer and technological conditions in spinning, drawing and preparation, as well as on the technology of textile processing and, in particular, physical and chemical treatments.

To give an example, the elastic recovery at different elongations for a Vectra polypropylene fibre is mentioned briefly below. Both high- and medium-tenacity types of this fibre show comparable elastic recovery giving

at 5 % elongation 80 % instant recovery and no residual deformation,
at 10 % elongation 95 % elastic recovery and 5 % residual deformation,

at 15 % elongation 90 % elastic recovery and 10 % residual deformation,
at 20 % elongation 17.5 % residual deformation.

The fibre exhibiting a very low tenacity of 200 mN tex^{-1}, produced under special conditions at high temperature, gives at 50 % elongation an instant elastic recovery of 95 % and recovers completely after 5 minutes.

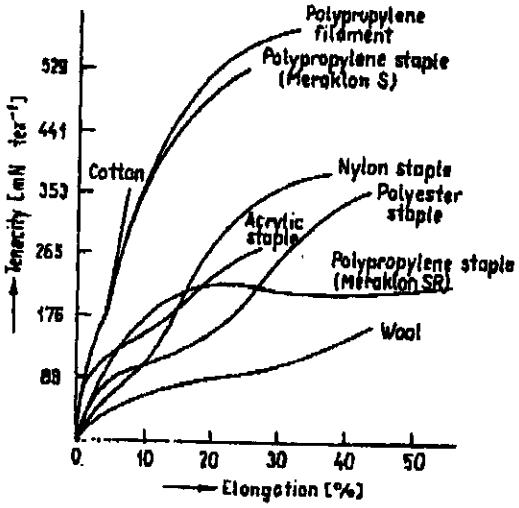

Fig. 24. Load-elongation curves for some fibres

In terms of elasticity polypropylene fibres are ranked below nylons. It should be pointed out that the recovery, and the secondary recovery in particular, of medium-tenacity polypropylene fibres is fairly slow and, in practice, the fibres are considered as only slightly lively.

2.3.3.4 Thermal properties and light exposure

Melting point: about 160–175 °C.
Softening point: about 140–150 °C.
Heat conductivity: 11.7×10^{-14} J cm^{-1} s^{-1} K^{-1}.
Specific heat: 1.9 J g^{-1} K^{-1}.

Polypropylene fibres show the lowest heat conductivity and, consequently, the highest insulating capacity of all commercial fibres. As for the heat conductivity, given as a relative index against air, some fibres in this respect are compared in Table 7.

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Table 7 – Heat conductivity of some textile fibres

Fibre	Index
(Air)	1.0
Polypropylene	2.8
Polyvinylchloride	6.4
Wool	7.4
Acetate	8.6
Viscose rayon	11.0
Cotton	17.5

At −40 °C the fibre preserves its flexibility and shows a higher tenacity and a somewhat lower elongation than at 20 °C. With the increasing temperature the fibre tenacity reduces, as shown in Fig. 25.

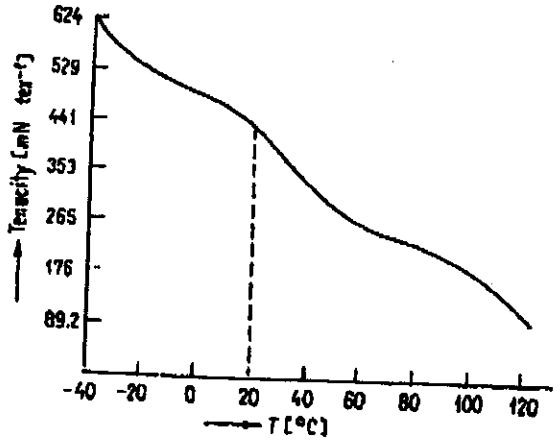

Fig. 25. Effect of temperature on the tenacity of polypropylene fibres

The fibre loses strength gradually with increases in temperature up to the point of softening. At the same time its elongation becomes somewhat higher and the course of the load-elongation relation changes. The zero strength is in the range of 160 – 165 °C. Heat-setting should be effected at about 130 – 135 °C.

Under excessive heat the fibre melts forming a molten droplet. In naked flame it melts and burns, and, detached from the flame, it extinguishes itself or burns only slowly. From the viewpoint of flammability the fibre behaviour is typical of polyolefine fibres.

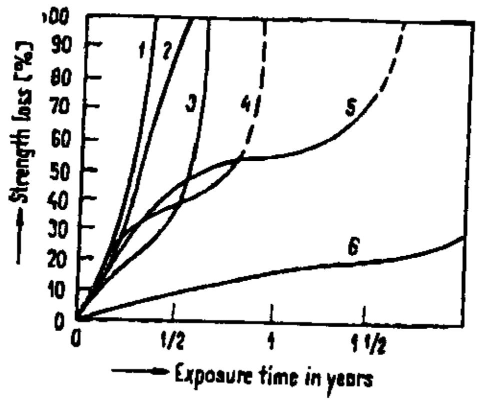

Fig. 26a. Time curves of the resistance of fibres exposed to natural weather conditions for two years in Wilmington in the U.S.A. 1 — silk, 2 — viscose rayon, 3 acetate, 4 — cotton, 5 — Cordura viscose rayon, 6 acrylic (Orlon)

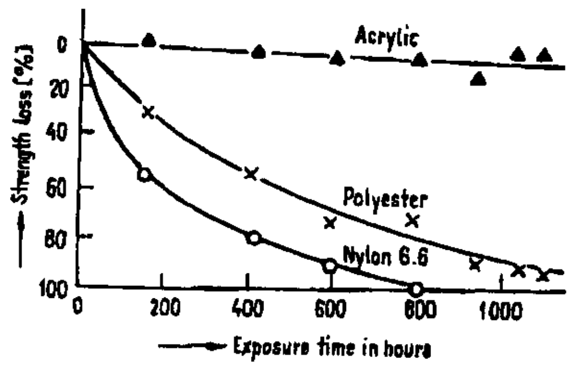

Fig. 26b. Resistance of acrylic, polyester and nylon 6.6 fibres to sunlight

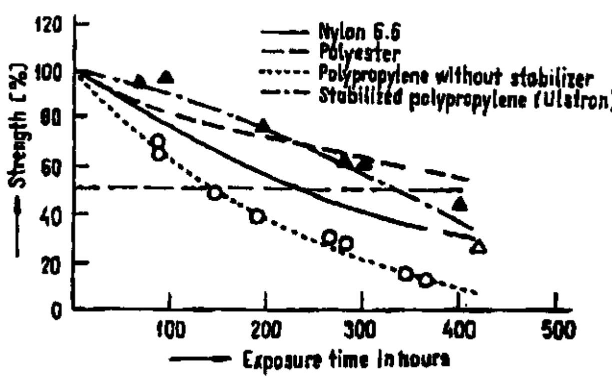

Fig. 26c. Resistance of polyester, nylon 6.6 and polypropylene fibres to weathering in South Florida in the U.S.A.

BROOKSTEIN DECLARATION EXHIBIT 13

Shakespeare, a world leader and producer of specialty monofilaments and polymers.

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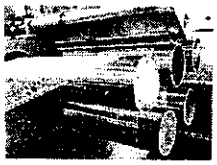

Specialty/High Performance Monofilaments

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PVDF Monofilament: Polyvinylidene Fluoride.

Composition:

- Melt Point = 174°C (345°F)
- Density = 1.78g/cc

Attributes:

- Outstanding resistance to weathering/UV exposure
- Outstanding chemical resistance
- Excellent soil release (low surface energy)
- Low coefficient of friction
- Lowest moisture regain
- High continuous use temperature (150 °C)
- Inherent Flam Resistance

Typical Properties- Trial Lots	MX229
Diameter	0.015
Denier	1850
Tensile Strength (lbs)	19.18
Tenacity (gdp)	4.71
Elongation at Break (%)	17.62
Loop Strength (lbs)	7.19
Loop Tenacity (gdp)	1.76
Loop Elongation (%)	5.5
Loop Impact Strength (ft-lbs/in)	70 (678g wt)
Common Knot Strength (lbs)	8.06
Common Knot Tenacity (gdp)	1.98

Common Knot Elongation (%)	11.4
Free Shrinkage at 132 °C/10 mins (%)	16.3
Boiling Water Shrinkage/5 mins (%)	9.13

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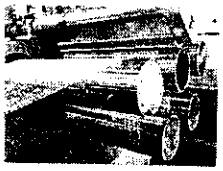

Polyolefin Monofilaments

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General Characteristics: Generally good chemical resistance. Good biological resistance.

Standard Products	Type	Breaking Strength* GPD	KPSI	Shrinkage in boiling water, %
MX-201	PP	4.0	46	11
MX-200	PP	3.0	35	6
MX-305	HDPE	4.2	50	12
MX-306	LDPE	1.7	21	57

PP = Polypropylene

HDPE = High Density Polyethylene

LDPE = Low Density Polyethylene

*GPD - Grams per denier
KPSI = Thousand pounds per square inch

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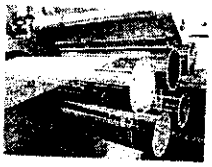
Polyester Monofilaments



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General Characteristics: Dimensionally stable, low moisture regain. Good UV and biological resistance.

Hydrolytically Stabilized Polyester: Specifically formulated to resist degradation caused by hot, moisture environments.

Sizes, shapes, colors...

Our monofilaments are available in a wide variety of shapes, colors and sizes depending on polymer types.

Various spool types are also available.

Standard Products	Breaking Strength* GPD	KPSI	Shrinkage at 200 °C, %
WP-550	4.5	80	6.5

Standard Polyester: For applications not requiring hydrolytic stability.

Standard Products	Breaking Strength* GPD	KPSI	Shrinkage at 200 °C, %
WP-104	4.5	80	3
WP-120	4.5	80	3
WP-200	6.0	107	15
WP-320	6.0	107	20

Superior abrasion resistant products.

*GPD - Grams per denier
KPSI = Thousand pounds per square inch

Shakespeare, a world leader and producer of specialty monofilaments and polymers.

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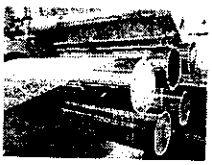

Nylon Monofilaments

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*GPD - Grams per denier
KPSI = Thousand pounds per square inch

General Characteristics: Tough, abrasion resistant.

Nylon 6: Flexible, resilient, resists compaction.

Standard Products	Breaking Strength* GPD	KPSI	Shrinkage at 175 °C, %
WN-18	6	88	6.5
WN-250	5.5	81	18
NX-1037*	5.5	81	6.5

* Low monomer for wet environments

Nylon 6,10: Low moisture regain. Increased chemical resistance. Improved dimensional stability.

Standard Products	Breaking Strength* GPD	KPSI	Shrinkage at 175 °C, %
WN-50	4.5	63	5
NX-201	5.5	76	12

Nylon 6,6: Higher operating temperature range.

Standard Products	Breaking Strength* GPD	KPSI	Shrinkage at 175 °C, %
WN-101	4.5	66	4
WN-125	5.5	81	3

NX-301	5.0	74	12

Both WN-125 and NX-301 are produced from a heat stabilized, high molecular weight

polymer which increases heat and abrasion.

BROOKSTEIN DECLARATION EXHIBIT 14

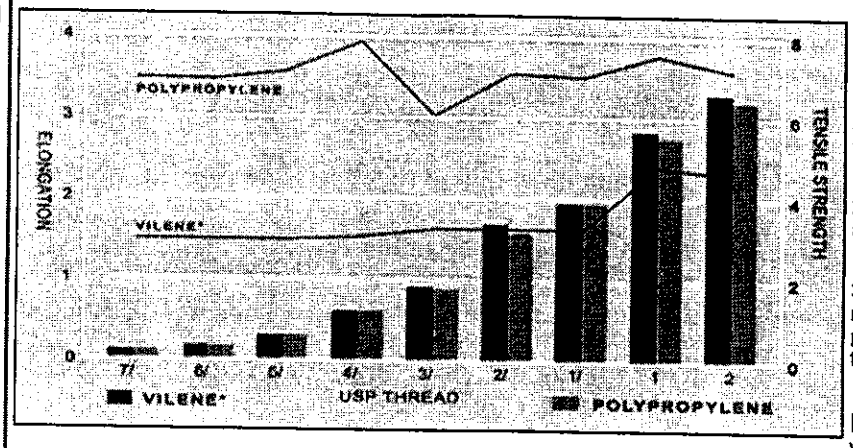
VILENE* - A MONOFILAMENT OF POLYVINYLIDENE FLUORIDE

BACK × HOME

Menu:
Main Pages
Fineline* Cutting
Reverse Cutting
Reverse Premium Cutting
Round Bodied Taper
A-Cute Taper*

❖ MONOFILAMENT P.V.D.F.

VILENE* is a monofilament of Polyvinylidene Fluoride (PVDF) which is non–absorbable physiologically inert. Maintaining it's tensile strength in situ, Vilene provides superior cli to the Surgeon. Combining the high tensile strength and low elongation (see chart belo excellent handling and knotting properties.



NOTES
1. Standard knot pull European Pharmaco US Pharmacopeia V absorbable sutures (for non-sterile suture

2. Standard knot pull a simple knot.

3. Elongation is the e material at break or r pull test expressed a the original length.

Experience has Vilene* has exc security. The springiness that affects all monofilaments when removed from the packag taken out by a gentle pull. It can be used in all procedures where polypropylene monofi utilised. Unlike polypropylene which after sterilisation is waxy in feel and is prone to fra is smooth, fray free and supple. Vilene* is sterilised by gamma irradiation. CV 300 neec cardiovascular sutures use a CV300 needle giving the Surgeon optimum performance suture material and needle.

Knotting:
As with all synthetic non-absorbable sutures, knot tying requires the standard surgical t flat and square ties with additional throws as indicated by surgical circumstance.
Vilene is available on the
following needles:

- ❖ Fineline Cutting
- ❖ Reverse Cutting
- ❖ Reverse Premium Point Cutting
- ❖ Round Bodied Taper
- ❖ Round Bodied A-Cute Taper

Both single & double armed sutures are available.

▲TOP

BROOKSTEIN DECLARATION EXHIBIT 15

FiberWire™

IMPORTANT PRODUCT INFORMATION
WICHTIGE PRODUKTINFORMATION
NOTICE D'UTILISATION IMPORTANTE
IMPORTANTI INFORMAZIONI PER L'USO
INSTRUCCIONES IMPORTANTES PARA EL USO

CE 0086

Arthrex

Manufacturer:
Arthrex Inc.
Naples, Florida 34108-1945 • USA
Toll-Free: +1 800 934-4404
www.arthrex.com

E. C. Representative:
Arthrex Med. Inst. GmbH
85757 Karlsfeld
Germany
Tel: +49 81 31 59 57 0 • Fax: +49 81 31 59 57 63 1

Arthrex

DFU-0065
Rev. 6

Description:
Arthrex FiberWire is available in several U.S.P. sizes (sutures meet U.S.P. standards for suture, except diameter). The Arthrex FiberWire may also be sold with needles attached (swaged) to the ends in a variety of sizes. The suture is made of polyethylene fibers and polyester fibers braided, sterilized and coated for surgical use. The coating acts as a lubricant for suture sliding, knot tying, and ease of passing suture through tissue. The Arthrex FiberWire is available non-dyed (white) or dyed and meets or exceeds U.S.P. and European standards (except for diameter).

Indications:
Arthrex FiberWire is indicated for use in soft tissue approximation and or ligation. FiberWire is not for use in cardiac indications.

Actions:
Arthrex FiberWire, when tested per ISO/DIS 10993, *Biological Evaluation of Medical Devices-Part 10: Tests for Irritations and Sensitization*, had no reactions of allergic or sensitive nature. The dyed suture and coating are pharmacologically inactive.

Arthrex FiberWire is not absorbed, but may become encapsulated in the surrounding connective tissues. The Arthrex FiberWire is not known to have significant change in tensile strength *in vivo*.

Contraindications:
None known

Warnings:
Do not re-sterilize. Once open, discard unused suture. Do not expose to heat.

Users should be familiar with surgical procedures and techniques involving non-absorbable sutures before employing Arthrex FiberWire for wound closure, as the risk of wound dehiscence may vary with the site of application and the suture material used.

As with any foreign body, prolonged contact of this or any other suture with salt solutions, such as those found in the urinary or biliary tracts, may result in calculus formations. Acceptable surgical practice must be followed with respect to drainage and closure of infected or contaminated wounds.

Precautions:
In handling this or any other suture material, care should be taken to avoid damage from handling. Avoid crushing or crimping damage due to application of surgical instruments such as forceps or needle holders.

Assure that all knots have been secured using accepted surgical knot tying techniques. Adequate knot security requires the accepted surgical technique of flat, square ties, with additional throws as warranted by surgical circumstance and the experience of the surgeon. The use of additional throws may be particularly appropriate when knotting monofilaments. Care should be taken to prevent damage to surrounding tissue or user puncture due to improper handling of the needlepoint.

Do not grasp the needle at the point or swage, to avoid damage to these areas. Reshaping needles may cause them to lose strength and be less resistant to bending and breaking. Discard used needles in "sharps" containers.

Adverse Reactions:
Adverse reactions have not been noted with the Arthrex FiberWire product in animal testing. Common non-absorbable suture reactions may include wound dehiscence, calculi formation in urinary and biliary tracts when prolonged contact with salt solutions such as urine and bile occurs, enhanced bacterial infectivity, minimal acute inflammatory tissue reaction, pain, edema, and erythema at the wound site. Inadvertent needle sticks with contaminated surgical needles may result in the transmission of bloodborne pathogens.

Sterilization:
Arthrex FiberWire suture is supplied sterile.
Method of sterilization - EO
Do not resterilize. Do not use if package is opened or damaged. Discard opened, unused sutures.

Storage Conditions:
Store below 25°C, away from moisture and direct heat. Do not use after expiration date.

How Supplied:
The Arthrex FiberWire is available in several U.S.P. sizes (sutures meet U.S.P. standards for suture, except diameter). The suture is supplied sterile in pre-cut lengths and in some cases with swaged needles. The Arthrex FiberWire is available in non-dyed (white) or dyed colors. The suture is made of polyethylene fibers and polyester fibers braided, sterilized and coated for surgical use. The coating acts as a lubricant for suture sliding, knot tying, and ease of passing suture through tissue.

SYMBOLS USED ON LABELING

- Do not reuse
- QTY – Quantity
- STERILE EO – Sterile unless the package is damaged or open. Method of sterilization - EO
- STERILE R – Sterile unless the package is damaged or open. Method of sterilization - gamma radiation
- LOT – Lot number
- See package insert
- CE – The product meets the essential requirements of Medical Device Directive 93/42 EEC.
- Use by - year & month

Beschreibung:
Arthrex FiberWire ist in verschiedenen USP-Größen erhältlich (das Nahtmaterial entspricht den USP-Normen für Nahtmaterial, mit Ausnahme des Durchmessers). Arthrex FiberWire ist unter Umständen auch mit an den Fadenenden befestigten (gesenkgeschmiedeten) Nadeln unterschiedlicher Größen erhältlich. Das Nahtmaterial besteht aus geflochtenen, sterilisierten und für den chirurgischen Gebrauch beschichteten Polyethylen und Polyesterfäden. Die Beschichtung fungiert als Fadengleitmittel und erleichtert die Knotenbildung und das Durchziehen des Fadens durch das Gewebe. Arthrex FiberWire ist ungefärbt (weiß) oder gefärbt erhältlich und entspricht oder übertrifft USP- und europäische Standards (mit Ausnahme des Durchmessers).

Anwendungsgebiete:
Arthrex FiberWire ist für Weichgewebeapproximation und/oder -ligation vorgesehen. FiberWire nicht für Kardio-Indikationen verwenden.

Funktionen:
Tests bei Arthrex FiberWire gemäß ISO/DIS 10993, *Biological Evaluation of Medical Devices - Part 10: Reiz- und Sensibilisierungstests* ergaben keine allergischen oder empfindlichen Reaktionen. Das gefärbte Nahtmaterial und die Beschichtung sind pharmakologisch inaktiv.

Arthrex FiberWire wird zwar nicht absorbiert, jedoch unter Umständen vom umgebenden Bindegewebe eingekapselt. Bei Arthrex FiberWire wurde *in vivo* keine signifikante Änderung der Zerreißfestigkeit festgestellt.

Gegenanzeigen:
Unbekannt

Warnhinweise:
Nicht resterilisieren. Unbenutztes Fadenmaterial nach dem Öffnen entsorgen. Von Hitze fernhalten.

Benutzer sollten vor dem Verschließen von Wunden mit Arthrex FiberWire mit den chirurgischen Prozeduren und Techniken vertraut sein, bei denen nicht-absorbierbarer Faden verwendet wird, da das Dehiszenzrisiko je nach Anwendungsstelle und verwendetem Fadenmaterial unterschiedlich ist.

Wie bei Fremdkörpern aller Art kann der längere Kontakt dieses oder jedes anderen Fadenmaterials mit Salzlösungen, (wie sie z.B. im Harn- und Gallentrakt vorhanden sind) zu Calculusbildung führen. Bei der Drainage und beim Schließen von infizierten oder kontaminierten Wunden sind die in der Chirurgie üblichen Praktiken zu beachten.

Vorsichtsmaßnahmen:
Bei der Handhabung dieses oder jedes anderen Fadenmaterials sorgfältig darauf achten, dass das Material nicht beschädigt wird. Schäden durch Zusammendrücken oder Abklemmen mit chirurgischen Instrumenten wie Zangen oder Nadelhaltern nach Möglichkeit vermeiden.

Sicherstellen, dass sämtliche Knoten gemäß den akzeptierten chirurgischen Knotenbildungstechniken sicher befestigt wurden. Voraussetzung für angemessene Knotenhaltbarkeit ist die Verwendung von flachen, quadratischen Schleifen mit zusätzlichen Verknotungen, je nach chirurgischer Situation und Erfahrung des Chirurgen. Besonders beim Verknoten von monofilen Fäden sind unter Umständen zusätzliche Verknotungen angebracht. Sorgfältig vorgehen, um Schäden am umgebenden Gewebe und Benutzerpunktierung durch falsche Handhabung der Nadelspitze zu vermeiden.

Die Nadel nicht an der Spitze oder am Gesenk festhalten, um eine Beschädigung dieser Bereiche zu vermeiden. Nadeln können durch Umformen an Stärke verlieren und gegen Verbiegen und Abbrechen weniger widerstandsfähig werden. Nadeln in entsprechend gekennzeichneten Behältern entsorgen.

Nebenwirkungen:
Bei Tierversuchen wurden bei der Verwendung von Arthrex FiberWire keine Nebenwirkungen festgestellt. Zu den bei nicht-absorbierbarem Faden üblichen Reaktionen zählen unter Umständen Dehiszenz, Calculusbildung in Harn- und Gallenwegen bei längerem Kontakt mit Salzlösungen (wie sie im Urin und in der Gallenflüssigkeit vorhanden sind), verstärkte Bakterieninfektion, minimale akute Gewebeentzündungen, Schmerzen, Ödeme und Erythema an der Wundstelle. Versehentliches Stechen mit kontaminierten chirurgischen Nadeln kann zur Übertragung von Blutpathogenen führen.

Sterilisation:
Arthrex FiberWire wird steril geliefert.
Sterilisationsmethode - EO.
Nicht resterilisieren. Bei beschädigter oder zuvor geöffneter Packung nicht verwenden. Offenes, unbenutztes Fadenmaterial entsorgen.

Lagerungsbedingungen:
Unter 25 °C trocken und fern von direkter Hitzeeinwirkung lagern. Nicht nach dem Verfallsdatum verwenden.

Lieferform:
Arthrex FiberWire ist in verschiedenen USP-Größen erhältlich (das Nahtmaterial entspricht den USP-Normen für Nahtmaterial, mit Ausnahme des Durchmessers). Das Fadenmaterial wird steril in vorgeschnittenen Längen und in manchen Fällen mit gesenkgeschmiedeten Nadeln geliefert. Arthrex FiberWire ist ungefärbt (weiß) und gefärbt erhältlich. Das Nahtmaterial besteht aus geflochtenen, sterilisierten und für den chirurgischen Gebrauch beschichteten Polyethylen und Polyesterfäden. Die Beschichtung fungiert als Fadengleitmittel und erleichtert die Knotenbildung und das Durchziehen des Fadens durch das Gewebe.

AUF DER VERPACKUNG VERWENDETE SYMBOLE

- Nicht wiederverwenden
- QTY – Quantität
- STERILE EO – Steril, solange die Verpackung ungeöffnet und unbeschädigt ist. Sterilisationsmethode - EO
- STERILE R – Steril, solange die Verpackung ungeöffnet und unbeschädigt ist. Sterilisationsmethode - Bestrahlung
- LOT – Chargenbezeichnung
- Siehe Packungsbeilage
- CE – Das Produkt entspricht den grundlegenden Anforderungen der Richtlinie des Rates über Medizinprodukte 93/42/EWG.
- Verwendbar bis Jahr und Monat

Description:
La suture Arthrex FiberWire existe en plusieurs tailles U.S.P. et est conforme aux normes U.S.P. s'appliquant aux sutures (diamètre excepté). La suture Arthrex FiberWire est également commercialisée avec des aiguilles serties de différentes tailles. Cette suture se compose de fibres de polyéthylène et de fibres de polyester tressées, stérilisées et traitées en surface pour les applications chirurgicales. Ce revêtement joue le rôle de lubrifiant pour faciliter le glissement du fil, le serrage des nœuds et le passage du fil à travers les tissus. La suture Arthrex FiberWire est disponible en blanc (non teinté) ou teintée, et elle est conforme aux normes européennes et normes U.S.P. (diamètre excepté).

Indications:
La suture Arthrex FiberWire est indiquée pour la ligature et le rapprochement des tissus mous. La suture Arthrex FiberWire n'est pas indiquée pour la chirurgie cardiaque.

Réactions:
Aucune réaction allergique ou sensible n'a été observée lors de la soumission de la suture Arthrex FiberWire au test exigé par la norme ISO/DIS 10993 / *Evaluation biologique des dispositifs médicaux, Partie 10 : Essais d'irritation et de sensibilisation*. La suture teintée et le traitement en surface de la suture sont pharmacologiquement inactifs.

La suture Arthrex FiberWire est non résorbable. Elle peut cependant être encapsulée par le tissu conjonctif. Selon les données disponibles, la résistance à la traction de la suture Arthrex FiberWire ne change pas de manière significative *in vivo*.

Contre-indications:
Aucune contre-indication connue.

Précautions d'emploi:
Ne pas stériliser à nouveau. Jeter toute suture non utilisée dont l'emballage a été ouvert. Ne pas exposer à la chaleur.

Tout praticien suturant une plaie avec la suture Arthrex FiberWire doit être familiarisé aux techniques chirurgicales recommandées pour les matériaux non résorbables, car le risque de déhiscence de la plaie varie selon le site de l'intervention et selon le type de suture employé.

Comme avec tout matériau exogène, un contact prolongé de cette suture ou de tout autre fil avec un fluide salin, comme ceux circulant dans les voies urinaires ou biliaires, peut conduire à la formation de calculs. Le praticien devra respecter les règles chirurgicales relatives au drainage et à la fermeture de plaies infectées ou contaminées.

Précautions d'emploi:
Comme avec toute autre suture, éviter d'abîmer le fil lors de sa manipulation. Ne pas écraser le fil avec des instruments chirurgicaux comme une pince ou un porte-aiguille.

Réaliser tous les nœuds conformément aux techniques chirurgicales en vigueur. Opter pour le nœud plat, qui garantit une bonne sécurité et qui est largement utilisé, avec boucles supplémentaires en fonction du cas chirurgical et de l'expérience du praticien. Si le fil est monofilament, prévoir des boucles supplémentaires pour les nœuds.

Bien contrôler la pointe de l'aiguille pour éviter de piquer les tissus environnants ou de blesser le praticien.

Ne pas saisir l'aiguille par sa pointe ou par son attache sur le fil pour éviter de l'endommager. Eviter de modifier la courbure des aiguilles pour ne pas réduire leur résistance à la déformation et à la rupture. Après usage, jeter les aiguilles dans un récipient spécial pour objets pointus et tranchants.

Effets indésirables:
Aucun effet indésirable particulier n'a été observé lors des tests du fil Arthrex FiberWire chez l'animal. Comme avec les autres fils de suture non résorbables, les réactions suivantes sont possibles : déhiscence de la plaie, formation de calculs dans les voies urinaires ou biliaires si contact prolongé avec des fluides salins comme l'urine ou la bile, infectivité bactérienne accrue, inflammation tissulaire minime, douleur, œdème et érythème au niveau de la plaie. Toute blessure avec une aiguille chirurgicale contaminée peut transmettre des germes pathogènes présents dans le sang.

Stérilisation:
La suture Arthrex FiberWire est livrée stérile.
Méthode de stérilisation : oxyde d'éthylène
Ne pas stériliser à nouveau. Ne pas utiliser si l'emballage est ouvert ou endommagé. Jeter les sutures non utilisées si leur emballage est ouvert.

Conditions de stockage:
Conserver à une température maximale de 25°C et à l'abri de l'humidité comme des sources de chaleur directes. Ne pas utiliser après la date d'expiration.

Présentation:
La suture Arthrex FiberWire existe en plusieurs tailles U.S.P. et elle est conforme aux normes U.S.P. s'appliquent aux sutures (diamètre excepté). La suture est livrée stérile en différentes longueurs précoupées. Elle est aussi disponible avec des aiguilles serties. La suture Arthrex FiberWire est disponible en blanc (non teinté) ou en couleurs (teinté). Cette suture se compose de fibres de polyéthylène et de fibres de polyester tressées, stérilisées et traitées en surface pour les applications chirurgicales. Ce revêtement joue le rôle de lubrifiant pour faciliter le glissement du fil, le serrage des nœuds et le passage du fil à travers les tissus.

SYMBOLES UTILISÉS SUR L'ÉTIQUETAGE

Ne pas réutiliser — QTY: Quantité

STERILE EO: Produit stérile si l'emballage n'a pas été ouvert ou endommagé. Méthode de stérilisation - EO

STERILE R: Produit stérile si l'emballage n'a pas été ouvert ou endommagé. Méthode de stérilisation - irradiation

LOT: No. de lot — Consulter la notice accompagnant le produit

CE: Ce produit est conforme aux exigences de la directive sur les dispositifs médicaux CEE 93/42.

À utiliser avant le (mois/année)

Descrizione:
FiberWire Arthrex è disponibile in molte misure U.S.P. (le suture soddisfano gli standard U.S.P. per sutura, tranne il diametro). FiberWire Arthrex può essere venduto anche con aghi di varie dimensioni attaccati (saldati) alle estremità. La sutura consiste in maglie di fibre di polietilene e di poliestere sterilizzate e rivestite per uso chirurgico. Il rivestimento funge da lubrificante per lo scorrimento della sutura, la chiusura dei nodi e la facilità di passaggio della sutura attraverso il tessuto. FiberWire Arthrex è disponibile sia non tinto (bianco) che tinto e soddisfa o supera gli standard U.S.P. ed europei (non per diametro).

Indicazioni:
FiberWire Arthrex è indicato per l'approssimazione e/o la legatura dei tessuti molli. FiberWire non va utilizzato in interventi cardiaci.

Azioni:
FiberWire Arthrex, quando testato per ISO/DIS 10993, *Valutazione biologica dei dispositivi medici-Parte 10: I test per irritazioni e sensibilizzazione*, non hanno evidenziato reazioni allergiche o ipersensibilità. La sutura e il rivestimento tinti sono farmacologicamente inattivi.

FiberWire Arthrex non viene assorbito, ma può essere incapsulato nei tessuti connettivi circostanti. Non sono noti per FiberWire Arthrex cambiamenti significativi nella resistenza alla tensione *in-vivo*.

Controindicazioni:
Nessuna nota

Avvertenze:
Non risterilizzare. Una volta aperte, gettare le suture non utilizzate. Non esporre al calore.

Gli utenti devono conoscere bene le procedure e le tecniche chirurgiche relative alle suture non assorbibili prima di utilizzare FiberWire Arthrex per la chiusura delle ferite, in quanto il rischio di deiscenza della ferita può variare in base al sito di applicazione ed al materiale utilizzato per la sutura.

Come per qualsiasi corpo estraneo, il contatto prolungato di questa e qualsiasi altra sutura con soluzioni saline, come quelle presenti nel tratto urinario o biliare, può risultare nella formazione di calcoli. È necessario seguire una pratica chirurgica corretta per il drenaggio e la chiusura di ferite infette o contaminate.

Precauzioni:
Nel trattare questo o qualsiasi altro materiale per sutura, occorre fare attenzione ad evitare danni dovuti al maneggiamento. Evitare danni da schiacciamento o piegature dovuti all'applicazione di strumenti chirurgici, inclusi forcipi o porta-aghi.

Assicurarsi che tutti i nodi siano stati legati usando le tecniche chirurgiche di annodatura accettate. Una sicurezza adeguata del nodo richiede la tecnica chirurgica accettata di legature piatte e quadrate, nonché di ulteriori avvolgimenti in base al caso chirurgico e all'esperienza del chirurgo. L'uso di avvolgimenti aggiuntivi può essere particolarmente appropriato per l'annodamento di monofilamenti. Evitare di recare danni al tessuto circostante o alla puntura dovuti ad una manipolazione non corretta della punta dell'ago.

Non afferrare l'ago per la punta o dalla saldatura, onde evitare danni a queste aree. Il riadattamento degli aghi può indebolirli e renderli meno resistenti alle piegature ed alle rotture. Gettare gli aghi usati in contenitori per materiale «affilato».

Effetti indesiderati:
Non si sono riscontrati effetti indesiderati con l'uso del prodotto FiberWire Arthrex nei test sugli animali. Le reazioni comuni alle suture non assorbibili possono includere la deiscenza della ferita, la formazione di calcoli nei tratti urinario e biliare dovuta al contatto prolungato con soluzioni saline come urina e bile, infettività batterica elevata, minima reazione del tessuto alle infiammazioni acute, dolore, edema ed eritema al sito della ferita. Il contatto involontario dell'ago con aghi chirurgici contaminati può risultare nella trasmissione di patogeni veicolati dal sangue.

Sterilizzazione:
La sutura FiberWire Arthrex viene fornita sterile.
Metodo di sterilizzazione - EO
Non risterilizzare. Non utilizzare se la confezione è aperta o danneggiata. Gettare le suture aperte non utilizzate.

Condizioni di conservazione:
Conservare al di sotto di 25°C, lontano da umidità e calore diretto. Non utilizzare dopo la data di scadenza.

Come si presenta:
FiberWire Arthrex è disponibile in molte misure U.S.P. (le suture soddisfano gli standard U.S.P. per sutura, tranne il diametro. La sutura viene fornita sterile in lunghezze pretagliate ed in alcuni casi con aghi saldati. FiberWire Arthrex è disponibile tinto o non tinto (bianco). La sutura consiste in maglie di fibre di polietilene e di poliestere sterilizzate e rivestite per uso chirurgico. Il rivestimento funge da lubrificante per lo scorrimento della sutura, la chiusura dei nodi e la facilità di passaggio della sutura attraverso il tessuto.

SIMBOLI USATI SULL'ETICHETTA

Monouso — QTY: Quantità

STERILE EO: Il prodotto è sterile se la confezione non è aperta o danneggiata. Metodo di sterilizzazione - EO

STERILE R: Il prodotto è sterile se la confezione non è aperta o danneggiata. Metodo di sterilizzazione - Raggi Gamma

LOT: Numero di lotto — Fare riferimento all'inserto contenuto nella confezione

CE: Il prodotto è conforme ai requisiti essenziali della Direttiva CEE 93/42 sui Dispositivi Medici.

Da usarsi entro anno e mese

Descripción:
La sutura FiberWire de Arthrex viene en varios tamaños aprobados por U.S.P. (las suturas cumplen las normas de U.S.P. para suturas, excepto en el diámetro). También es posible encontrar la sutura FiberWire de Arthrex en diversos tamaños y con agujas incorporadas (enhebradas) en los extremos. La sutura está hecha de fibras de polietileno y poliéster trenzadas, esterilizadas y recubiertas para uso quirúrgico. El recubrimiento hace las veces de lubricante para deslizar la sutura, atar los nudos y facilitar el paso de la sutura a través del tejido. La sutura FiberWire de Arthrex viene en modelos sin teñir (blanca) o teñida y cumple o supera las normas de U.S.P. y Europa (excepto en el diámetro).

Indicaciones:
La sutura FiberWire de Arthrex está indicada para aplicaciones de aproximación y ligadura de tejido blando. La sutura FiberWire no está indicada para uso cardíaco.

Acciones:
Sutura FiberWire de Arthrex, sometida a prueba de acuerdo con la norma ISO/DIS 10993, *Evaluación Biológica de Dispositivos Médicos-Sección 10: En pruebas para detección de irritaciones y sensibilidad*, no hubo reacciones alérgicas ni de sensibilidad. La sutura teñida y el recubrimiento son inactivos farmacológicamente.

La sutura FiberWire de Arthrex no se absorbe, pero podría encapsularse en los tejidos conjuntivos adyacentes. La sutura FiberWire de Arthrex no presenta cambios significativos conocidos en cuanto a resistencia a la tracción *in vivo*.

Contraindicaciones:
Ninguna conocida

Advertencias:
No esterilizar de nuevo. Una vez abierto el paquete, desechar la sutura no utilizada. No exponer al calor.

Los usuarios deberán conocer los procedimientos quirúrgicos y las técnicas con suturas no absorbibles antes de utilizar FiberWire de Arthrex para cerrar heridas, ya que el riesgo de dehiscencia de las heridas varía según el sitio de aplicación y el material de sutura utilizado.

Al igual que ocurre con todo cuerpo extraño, el contacto prolongado de esta sutura, o de cualquier otro tipo de sutura, con soluciones salinas como las halladas en los tractos urinario o biliar, podría producir cálculos. Se deben usar métodos quirúrgicos aceptables en relación con el drenaje y cierre de heridas infectadas o contaminadas.

Precauciones:
Se debe tener cuidado al manipular esta o cualquier otro material de sutura para evitar dañarlo. No utilice instrumentos quirúrgicos o de aplicación tales como fórceps o porta-agujas para evitar aplastar o plegar el material.

Cerciórese de que todos los nudos se hayan fijado por medio de técnicas aceptadas para nudos quirúrgicos. Para la fijación correcta de los nudos es necesario utilizar la técnica quirúrgica aceptada de nudos planos y cuadrados con lazadas adicionales, según lo requieran las condiciones quirúrgicas y la experiencia del cirujano. El uso de lazadas adicionales podría ser especialmente útil en la elaboración de nudos de monofilamentos. Debe tenerse cuidado para evitar daños al tejido adyacente o punciones producidas por el usuario al manipular incorrectamente la punta de la aguja.

No sujete la aguja por la punta ni por el ojo para evitar daños en esas partes. Si se modifica la forma de las agujas, éstas podrían perder su firmeza y ser menos resistentes a las curvaturas y el rompimiento. Deseche las agujas usadas en recipientes para objetos punzantes.

Reacciones adversas:
No se han detectado reacciones adversas del producto FiberWire de Arthrex en pruebas con animales. Entre las reacciones comunes de las suturas no absorbibles se encuentran: dehiscencia de las heridas, formación de cálculos en los tractos urinario y biliar en condiciones de contacto prolongado con soluciones salinas tales como la orina y la bilis, mayor propensión a infecciones bacterianas, reacción mínima inflamatoria aguda del tejido, dolor, edema y eritema en el sitio de la herida. El pinchazo accidental con agujas quirúrgicas usadas podría causar la transmisión de patógenos a través de la sangre.

Esterilización:
La sutura FiberWire de Arthrex se suministra estéril.
Método de esterilización - EO
No esterilizar de nuevo. No utilizar si el paquete llega abierto o dañado. Desechar las suturas abiertas que no se hayan utilizado.

Condiciones de almacenamiento:
Almacenar el producto por debajo de 25° C, alejado de la humedad y el calor directo. No utilizar después de la fecha de caducidad.

Presentación:
La sutura FiberWire de Arthrex viene en varios tamaños aprobados por U.S.P. (las suturas cumplen las normas U.S.P. para sutura, excepto en el diámetro). La sutura se suministra estéril en cortes de longitud predeterminada y en algunos casos con agujas enhebradas. La sutura FiberWire de Arthrex se encuentra disponible en modelos sin teñir (blanca) o teñida. La sutura está hecha de fibras de polietileno y poliéster trenzadas, esterilizadas y recubiertas para uso quirúrgico. El recubrimiento hace las veces de lubricante para deslizar la sutura, atar los nudos y facilitar el paso de la sutura a través del tejido.

SIMBOLOS UTILIZADOS EN LAS ETIQUETAS

No reutilizar — QTY: Cantidad

STERILE EO: Estéril mientras el envase no sea abierto o dañado. Método de esterilización - EO

STERILE R: Estéril mientras el envase no sea abierto o dañado. Método de esterilización - irradiación

LOT: Número de lote — Ver folleto en el interior del paquete

CE: Este producto cumple con las normas básicas de la Directiva de productos médicos, 93/42 CEE

Caduca - año y mes